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# TABLE OF CONTENTS

Vol. 27 December 1, 1944 No. 12

## THE JOURNAL

Notes on Moisture Expansion ..... 355–58
By H. Thiemecke

Cone 01 Glazes for Low-Temperature Vitreous Ware ..... 358–66
By John Marquis

Abrasion Resistance of Porcelain Enamels ..... 366–69
By Clark Hutchison

Effect of Fluorine and Phosphorus Pentoxide on Properties of Soda-Dolomite Lime-Silica Glass ..... 369–72
By Owens-Illinois Glass Company General Research Laboratory

Refined Method of Control of Cordiness and Workability of Glass During Production, Statistical Control Chart Applied to Daily Measurements of Density ..... 373–87
By L. G. Ghering

Author Index to *The Journal* ..... 389–90

Subject Index to *The Journal* ..... 390–96

Footnote Citations Index to *The Journal* ..... 396–401

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# NOTES ON MOISTURE EXPANSION*

By H. Thiemecke

Abstract

Moisture-expansion determinations were made over a period of more than three years on bisque and glazed specimens of two types of semivitreous bodies having approximately the same absorption which were exposed to four different atmospheric conditions. The data indicate the cone 6 talc-pyrophyllite body to expand less under all conditions of exposure than the cone 9 clay-flint-feldspar body. Glazed specimens of both bodies expanded more than the bisque specimens and a possible explanation is given. From mathematical considerations, a three-year storage period appears to be sufficient to permit moisture expansion that closely approaches the ultimate which might be expected in the types of bodies investigated. The autoclave treatment gave the highest expansion values and should serve as an index of the service performance as to delayed crazing of ware so tested.

## I. Introduction

Of the various reports[1] on moisture expansion of ceramic bodies, none includes data covering the rate of such expansion shortly after removal from the kiln. To supply such information in part, moisture-expansion determinations were made at various intervals on specimens of two types of semivitreous dinnerware bodies stored under various atmospheric conditions after firing.

## II. Bodies and Methods

The investigation covered a cone 9 clay-flint-feldspar body and a cone 6 body composed of clay, flint, feldspar, talc, and pyrophyllite. The absorption values of the bisque bodies were 9.0% and 9.3%, respectively. The methods of preparation of the specimens and expansion determinations were similar to those reported in a previous investigation[1(l)] on moisture expansion. Half of the cone 9 body specimens were covered with a cone 5 glaze and half of the cone 6 body specimens with a cone 02 glaze. To maintain the same heat-treatment for both types of specimens, the unglazed rods were fired with their respective glazed ones; but to obviate contamination by the vola-

* Presented at the Forty-Sixth Annual Meeting, The American Ceramic Society, Pittsburgh, Pa., April 5, 1944 (White Wares Division). Received September 9, 1944.

[1] (*a*) H. G. Schurecht, "Methods for Testing Crazing of Glazes Caused by Increase in Size of Ceramic Bodies," *Jour. Amer. Ceram. Soc.*, **11** [5] 271–77 (1928).

(*b*) H. G. Schurecht and G. R. Pole, "Effect of Water in Expanding Ceramic Bodies of Different Compositions," *ibid.*, **12** [9] 596–604 (1929).

(*c*) H. G. Schurecht and G. R. Pole, "Methods of Measuring Strains Between Glazes and Ceramic Bodies," *ibid.*, **13** [6] 360–75 (1930).

(*d*) H. G. Schurecht and G. R. Pole, "Moisture Expansion of Glazes and Other Ceramic Finishes," *ibid.*, **14** [4] 313–18 (1931).

(*e*) E. P. Wright, "Study of Absorption of Two Earthenware Bodies and Their Resistance to Crazing in Steam Test," *ibid.*, **12** [2] 118–22 (1929).

(*f*) R. G. Mills, "Use of Autoclave in Testing Ceramic Products for Resistance to Crazing," *ibid.*, **13** [12] 903–14 (1930).

(*g*) H. H. Holscher, "Effects of Autoclave Treatments on Ceramic Bodies and Clay" *ibid.*, **14** [3] 207–18 (1931).

(*h*) H. E. Davis and R. L. Lueders, "Simplification of Ring Method for Determining Glaze Stresses," *ibid.*, **15** [1] 34–36 (1932).

(*i*) E. H. Lintz, "Use of Talc and Pyrophyllite in Semivitreous Dinnerware Bodies," *ibid.*, **21** [6] 229–37 (1938).

(*j*) R. F. Geller and A. S. Creamer, "Note on Moisture Expansion of Ceramic Whiteware in Storage and in Service," *ibid.*, **24** [3] 77–79 (1941).

(*k*) W. E. Palmer, "Influence of Flint Particle Size on Permanent Moisture and Thermal Expansion in Porous Earthenware Bodies," *ibid.*, **25** [14] 413–16 (1942).

(*l*) H. Thiemecke, "Thermal and Moisture Expansion of Kaolins and Bodies at Different Temperatures," *ibid.*, **24** [2] 69–75 (1941).

(*m*) H. Thiemecke, "Thermal and Moisture Expansion of Ball Clays and Bodies at Different Temperatures," *ibid.*, **26** [6] 173–79 (1943).

(*n*) H. H. Holscher, "Expansion of Ceramic Bodies Caused by Liquid and Vapor Penetration," *Ohio State Univ. Eng. Expt. Station Circ.*, No. 22, 24 pp. (1931); *Ceram. Abs.*, **16** [2] 68 (1937).

(*o*) R. F. Geller and A. S. Creamer, "Moisture Expansion of Ceramic Whiteware," *Bur. Stand. Jour. Research*, **9** [3] 291–307 (1932); RP 472; *Ceram. Abs.*, **11** [12] 622 (1932).

TABLE I

PERCENTAGE MOISTURE EXPANSION OF CONE 6 TALC-PYROPHYLLITE BODY EXPOSED TO DIFFERENT CONDITIONS

| Time (days) | Air exposed (%) | | Over $H_2SO_4$ (%) | | $H_2O$ submerged (%) | | Steam autoclaved (%) | |
|---|---|---|---|---|---|---|---|---|
| | Bisque | Glazed | Bisque | Glazed | Bisque | Glazed | Bisque | Glazed |
| 2/24 | 0.002 | 0.003 | 0.000 | 0.002 | 0.023 | 0.033 | 0.064 | 0.083 |
| 7/24 | .006 | .023 | .000 | .011 | .028 | .047 | | |
| 19/24 | .011 | .039 | .000 | .020 | .030 | .055 | | |
| 1 | .014 | .042 | .000 | .023 | .031 | .056 | | |
| 2 | .017 | .047 | .000 | .029 | .033 | .059 | | |
| 3 | .018 | .049 | .000 | .033 | .034 | .061 | | |
| 4 | .018 | .051 | .000 | .033 | .035 | .062 | | |
| 7 | .019 | .052 | .000 | .033 | .037 | .064 | | |
| 14 | .020 | .053 | .000 | .033 | .039 | .066 | | |
| 21 | .020 | .053 | .000 | .033 | .041 | .068 | | |
| 42 | .022 | .055 | .000 | .033 | .044 | .069 | | |
| 63 | .023 | .057 | .000 | .033 | .046 | .071 | | |
| 93 | .025 | .058 | .000 | .033 | .048 | .072 | | |
| 155 | .027 | .061 | .000 | .033 | .050 | .073 | | |
| 365 | .031 | .063 | .000 | .033 | .055 | .077 | | |
| 803 | .039 | .067 | .000 | .033 | .061 | .079 | | |
| 1160 | .042 | .069 | .000 | .033 | .062 | .081 | | |

tils of the glaze, the unglazed rods were housed in a tightly fitting refractory container. After removal from the kiln at around 400°C., the specimens were cooled in a desiccator over concentrated sulfuric acid, quickly measured, and equal portions were (1) exposed to the laboratory atmosphere, (2) submerged in distilled water, (3) stored in a desiccator over concentrated sulfuric acid, and (4) autoclaved for three hours at 150 lb. steam pressure. Except in the case of (4), expansion determinations were made at various intervals over a period of more than three years.

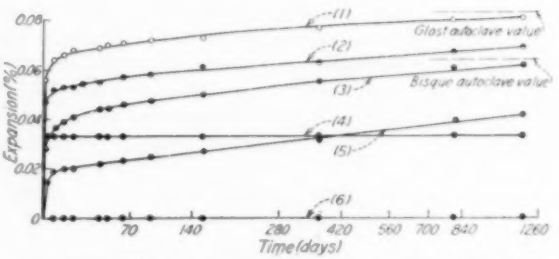


FIG. 1.—Moisture expansion of cone 6 talc-phyrophyllite semivitreous body; *curve* (1), glazed specimens water submerged; *curve* (2) glazed specimens in atmosphere; *curve* (3) bisque specimens water submerged; *curve* (4) glazed specimens in desiccator; *curve* (5) bisque specimens in atmosphere; *curve* (6) bisque specimens in desiccator.

## III. Results and Discussions

Values for the moisture expansion obtained under the foregoing conditions are given in Tables I and II and Figs. 1 and 2. The data indicate the clay-flint-feldspar body to show a substantially higher expansion in the presence of water than the talc-pyrophyllite body. The rate of this expansion is also higher for the clay-flint-feldspar body. As expected, the initial rate of expansion of both bodies was much greater when submerged in water than when exposed to air. After this initial expansion, however, the rates of expansion of the water-submerged and air-exposed specimens approached each other more closely and those for the talc-pyrophyllite body became almost identical. The rapid initial rehydration of the air-exposed specimens is significant, indicating great avidity for moisture. Of further significance is the asymptotic approach of the expansion of the water-submerged specimens to the steam autoclave values. Since from a moisture-expansion standpoint water submersion represents the most severe service condition a semivitreous ware might encounter, the more severe autoclave test developed by Schurecht[1(a)] in his pioneering work can be accepted as an index of the service performance that might be expected of ware so tested. That the expansion due to the water submersion would ever equal that obtained in the autoclave is problematical, however, since a steam pressure of 150 lb. requires a temperature of 183°C.; this is sufficient to produce small voids owing to the unequal expansion and contraction along different axes of the unoriented cristobalite particles present. This dilatation is additive to that caused by the steam hydration.

The tendency of the clay-flint-feldspar body to expand more in the presence of even low-moisture concentrations is shown in the results obtained on the specimens held over concentrated sulfuric acid. Under these conditions, the talc-pyrophyllite body unglazed rods showed no expansion whereas the clay-flint-feldspar bisque rods showed a slight expansion resulting either from the water vapor ever present over sulfuric acid or from that encountered during the short exposures to the air when the specimens were removed from the desiccator for measuring.

The higher expansion under all exposure conditions of the glazed rods of both bodies is interesting. This increased expansion was at first thought to be due to the alkali which may have volatilized into the body from the glaze, but the consistently higher expansion of the glazed rods in the desiccated atmosphere precluded this assumption. An explanation was then sought in the difference between the thermal expansions of the bodies and glazes and the plastic properties of the glazes. Since both bodies had a higher thermal expansion than the respective glazes with which they were covered (thermal expansion 25° to 600°C. of cone 9 body was 0.53%; cone 5 glaze, 0.38%; cone 6 body,

TABLE II

PERCENTAGE MOISTURE EXPANSION OF CONE 9 SEMIVITREOUS BODY EXPOSED TO DIFFERENT CONDITIONS

| Time (days) | Air exposed (%) | | Over $H_2SO_4$ (%) | | $H_2O$ submerged (%) | | Steam autoclaved (%) | |
|---|---|---|---|---|---|---|---|---|
| | Bisque | Glazed | Bisque | Glazed | Bisque | Glazed | Bisque | Glazed |
| 1/24 | 0.012 | 0.003 | 0.000 | 0.002 | 0.030 | 0.053 | 0.145 | 0.174 |
| 7/24 | .023 | .018 | .000 | .005 | .040 | .080 | | |
| 19/24 | .028 | .042 | .000 | .011 | .043 | .088 | | |
| 1 | .030 | .047 | .000 | .014 | .045 | .090 | | |
| 2 | .033 | .055 | .000 | .021 | .047 | .093 | | |
| 3 | .035 | .059 | .000 | .027 | .049 | .095 | | |
| 4 | .035 | .062 | .000 | .027 | .052 | .097 | | |
| 7 | .037 | .068 | .000 | .027 | .055 | .098 | | |
| 14 | .039 | .071 | .000 | .027 | .059 | .103 | | |
| 21 | .043 | .072 | .000 | .027 | .064 | .107 | | |
| 28 | .046 | .075 | | | | | | |
| 42 | .049 | .080 | .002 | .027 | .069 | .112 | | |
| 49 | .049 | .082 | .002 | .027 | .074 | .114 | | |
| 63 | .052 | .084 | .004 | .028 | .078 | .118 | | |
| 91 | .058 | .088 | | | .083 | .125 | | |
| 119 | .064 | .092 | | | .087 | .128 | | |
| 154 | .066 | .092 | .004 | .030 | .095 | .130 | | |
| 365 | .078 | .103 | .006 | .031 | .113 | .146 | | |
| 803 | .091 | .113 | .009 | .034 | .133 | .164 | | |
| 1160 | .096 | .118 | .010 | .036 | .143 | .172 | | |

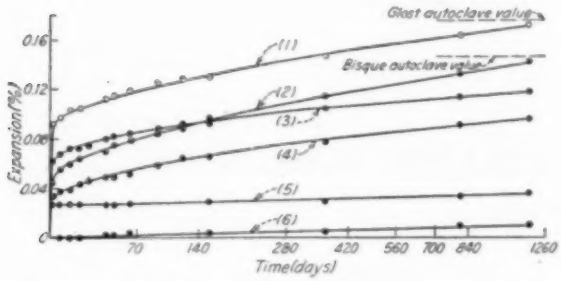


FIG. 2.—Moisture expansion of cone 9 semivitreous body; *curve* (1) glazed specimens water submerged; *curve* (2) bisque specimens water submerged; *curve* (3) glazed specimens in atmosphere; *curve* (4) bisque specimens in atmosphere; *curve* (5) glazed specimens in desiccator; *curve* (6) bisque specimens in dessicator.

0.62%; cone 02 glaze, 0.40%) in cooling from the transformation points of the glazes, the rods might be considered to have compressed the more or less still plastic glazes into ridges. As the glazes became more rigid with time at room temperature and began to straighten out, the rods were placed in tension and elongated. In an effort to substantiate this view, several additional rods were covered with a high-expansion glaze of high alkali content (which was finely crazed on removal from the kiln) and under the various conditions of exposure imposed were found to have practically the same expansion as the unglazed rods.

## IV. Mathematical Considerations

Granting that the autoclave value represents the maximum moisture expansion obtainable in a porous ceramic body, it would be desirable to know also the ultimate expansion that would obtain under storage in the atmosphere. Assuming that the expansion is directly dependent on the weight of water taken up by the body either by absorption or chemical combination, if the weight of water taken up in time $t$ is $y$ and if $m$ is the maximum weight of water taken up in storage, then

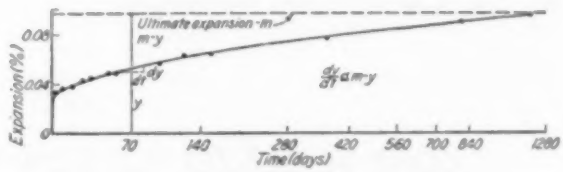


FIG. 3.—Relation between observed and ultimate expansion of cone 9 semivitreous body exposed to atmosphere.

the velocity of expansion may be represented by the proportionality (1). This may be expanded to equation (2) and on clearing yields equation (3).

$$\frac{dy}{dt} \alpha (m - y) \tag{1}$$

$$\frac{\frac{dy_1}{dt_1}}{\frac{dy_2}{dt_2}} = \frac{m - y_1}{m - y_2} \tag{2}$$

$$m = \frac{\frac{dy_1}{dt_1} y_2 - \frac{dy_2}{dt_2} y_1}{\frac{dy_1}{dt_1} - \frac{dy_2}{dt_2}} \tag{3}$$

$$\frac{dy}{dt} = k(m - y) \tag{4}$$

$$y = m(1 - e^{-kt}) \tag{5}$$

It is thus possible in Fig. 3, in which is plotted the expansion data obtained on the cone 9 clay-flint-feldspar bisque specimens stored in the atmosphere, to determine the maximum expansion, $m$, by taking several values of $y$ and measuring the tangents at these points. The accuracy of this determination obviously is influenced by the accuracy involved in measuring the tangents at the two $y$ points and may be enhanced by using a number of $y$ values and then averaging the various $m$ values obtained. By placing proportionality (1) into the equation form (4) and then integrating, the

exponential equation (5) may be derived in which $k$ may be regarded as the fraction of the ultimate expansion taking place in unit time. In an exponential process such as this, the accuracy of the $m$-value determination may be checked by plotting log $(m - y)$ against time, since for the correct value of $m$ a straight line should result.[2] Considering the case of the cone 6 and cone 9 bisque specimens exposed to the atmosphere whose expansion is not complicated by that of the glaze, the calculated values for $m$ were 0.044% and 0.097%, respectively. These values closely agree with those obtained experimentally covering an exposure of over three years and indicate that after this length of exposure not much change may be expected in the moisture expansion of the two types of bodies studied.

## V. Summary

Of two types of semivitreous dinnerware bodies having approximately the same absorption, which were investigated for a period of over three years, the cone 6 talc-pyrophyllite body showed the least moisture expansion under various conditions of storage. Both bodies apparently have a strong affinity for water as is evidenced by the rapid initial expansion which later approached an ultimate value asymptotically. A three-year storage period appears to be sufficient to give expansion values which closely approach the ultimate that may be expected.

The autoclave treatment gave maximum expansion values and such exposure should indicate the resistance of ware so tested to delayed crazing due to moisture expansion.

Specimens covered with glazes of low thermal expansion dilated more than the corresponding bisque specimens but, when covered with a high thermal-expansion glaze which crazed, they showed approximately the same moisture expansion as the bisque rods, indicating thereby the partial effect of stresses that occur due to differences between the thermal expansions of a body and glaze.

[2] F. H. Getman, Outlines of Theoretical Chemistry, p. 435. John Wiley & Sons, Inc., New York, 1928.

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# CONE 01 GLAZES FOR LOW-TEMPERATURE VITREOUS WARE*

By John Marquis

Abstract

The production of two-fired vitreous ware using a nepheline syenite body biscuited at cone 4 requires a glaze of low expansion maturing at cone 01 or below. Such a glaze can be produced using not more than 0.05 KNaO and up to 0.19 MgO in the empirical formula if 70 to 80% of the glaze batch is fritted. Such glazed ware showed good resistance to crazing, and modulus of rupture and scratch-hardness values were equal to commercial hotel china.

## I. Introduction

This investigation was established to produce a glaze similar in some respects to the dinnerware glazes now used but incorporating several unique properties involving maturing temperature, thermal expansion, hardness, and effect on underglaze colors which specially adapt it for use on a vitreous body containing from 54 to 60% nepheline syenite. The maturing temperature at cones 02 to 01 is five cones below that of the body and provides a safe range for two-fire ware to eliminate any unnecessary warpage in the glost fire.

A standard cone 4 dinnerware glaze which exhibits good fit on hotel china will often have poor craze resistance on the syenite body glost fired to cone 01. Evidence for this fact is shown by the glazed-ring test in which a compression of over 0.05 mm. exists on the former type ware but only 0.018 mm. compression on the latter ware. For good glaze fit at cone 01, therefore, the thermal expansion of the standard glaze must be lowered by increasing those oxides with low expansion and decreasing those with high expansion.

In fulfilling these two objectives, the glaze should not lose any of the properties of scratch hardness and resistance to acid attack found in commercial hotel china glazes. There should be no substantial increase in those oxides, such as $K_2O$, $Na_2O$, PbO, and $B_2O_3$, which produce "soft" glazes.

Because a body of this type lends itself to underglaze colors, zinc and zincless glazes were produced to provide an opportunity for a wide range of colors.

## II. Procedure

### (1) General

The research has been conducted in two phases as follows: (1) The laboratory investigation of possible glaze ingredients was made, and the best combination of the RO oxides was determined by two triaxial studies. (2) A plant trial was made in a commercial cone 02 tunnel kiln of the best glazes, and the resulting ware

* Presented at the Forty-Sixth Annual Meeting, The American Ceramic Society, Pittsburgh, Pa., April 5, 1944. Received September 15, 1944.

was subjected to the following tests: modulus of rupture, glazed-ring, thermal shock, autoclave, impact, chipping, scratch hardness, and frit solubility.

## (2) Specific

(*A*) *Bodies:* The experimental glazes were applied to vitreous syenite bodies, similar in composition to those developed by Koenig.[1] Table I gives the composition of three of these bodies which vary only in the choice of ball clays employed. In the laboratory, ware similar to No. 11 body was used. Five-inch plates, rings, and bars all made from No. 10 body were used in the first plant trial. The second plant trial was made on 5-in. plates jiggered from No. 13 body.

TABLE I

COMPOSITION OF THREE NEPHELINE SYENITE BODIES

| Material | No. 10 body | No. 11 body | No. 13 body |
|---|---|---|---|
| Nepheline syenite | 54 | 54 | 54 |
| Flint | 6 | 6 | 6 |
| Edgar plastic kaolin | 8 | 8 | 8 |
| Harris Lunday kaolin | 8 | 8 | 8 |
| Pioneer Ga. kaolin | 8 | 8 | 8 |
| Old Mine ball clay No. 4 | 16 | | 8 |
| Bell dark ball clay | | 16 | 8 |

(*B*) *Tests:* The testing of the physical properties of the glazes was conducted according to the following procedures:

(i) *Modulus of Rupture:* Bars were cast from molds 7 in. long and 1 sq. in. in cross section. The average modulus of rupture was determined on both glazed and unglazed fired bars.

(ii) *Glazed-Ring Test:* Rings were cast which when fired measured $2^3/_4$ in. in diameter, $^5/_8$ in. high, and $^1/_4$ in. in width of the band. Glaze was applied only to the exterior circumference, the top, bottom, and interior being left bare. After the glost fire, two brass tabs were applied to the top of the band $^3/_{16}$ in. apart. Reference points were scratched on both tabs, and the distance between them was measured with a microscope equipped with a filar head so that readings could be checked to $\pm 0.004$ mm. The ring was sawed in between the two tabs and the measurement again taken. A closing up of the ring indicated glaze compression and and opening up indicated tension.

(iii) *Autoclave:* The autoclave is primarily a test for semivitreous ware in which moisture expansion can occur. Since the test plates in the first plant trial had an absorption of over 2%, the autoclave gave significant results of the craze resistance of the various glazes. The test, run for one hour at 150 lb. per sq. in., was reached after a preliminary hour in which the pressure was increased 25 lb. every ten minutes and was followed by an hour's decrease of pressure at the same rate.

(iv) *Thermal Shock:* Two thermal-shock tests were employed. The first one consisted of quenching the ware in water at 25°C. after it had been in a thermostatically controlled oven at 200°C. for a one-hour period. The second test was conducted in the same fashion except that the oven temperature was at 175°C. The successful passage of five cycles in the latter test insures good glaze fit.[2] The former test is more severe and was used to obtain comparative values among the glazes.

(v) *Impact:* The impact test was conducted by dropping a 1-ounce ball on the center of the test plate from steadily increasing heights. When the blow was sufficient to cause the glaze to star on the bottom, the height of the fall was recorded, the plate was broken, and the thickness at the point of the star was measured with micrometer calipers to 0.001 in. The impact value was determined from formula (1).

$$\text{Impact value} = \frac{\text{height of drop (in.)}}{16 \times \text{plate thickness (in.)}} \qquad (1)$$

(vi) *Chipping:* The chipping of the edges of the plates was executed using a 5-ounce hammer which swung as a pendulum along a scale calibrated in foot-pounds. The chipping value is the foot-pound reading made by the blow which chipped the plate.

(vii) *Scratch Hardness:* Specimens of the various glazes were cemented on slides and scratched with a microcharacter using a 9-gm. weight on the diamond point. The width of the scratches was measured with a microscope equipped with a filar head, making it possible to check readings to $\pm 0.03$ micron.

(viii) *Solubility of Frits:* The various frits were ground; only that portion was used which passed the 100-mesh screen but was held by the 150-mesh screen. Fines were removed by several washings with alcohol. A 2.000-gm. sample was boiled for thirty minutes in 60 cc. of a 4% acetic acid solution in a 250-cc. Erlenmeyer flask equipped with a reflux condenser. At the end of the period, the solution was filtered through a previously dried and weighed Gooch crucible. After the frit had dried to constant weight in the crucible, the loss in weight was recorded. The value in frit solubility was this loss in weight taken as a percentage of 2.000 gm.

(*C*) *Visual Glaze Properties:* All glazes were examined for gloss, surface texture, and the appearance of any blemish. The following code was used to record the findings:

(i) *Gloss:* (*a*) *High*, glazes whose gloss was as brilliant as the best glazes found in the market; (*b*) *medium*, glazes with lower gloss but still equaling ware in the market; and (*c*) *low*, below-market standards for bright glazes.

(ii) *Surface Texture:* (*a*) *Excellent*, glazes whose smoothness equals the best glaze found in the market; (*b*) *good*, glazes with more obvious surface characteristics but still equaling commercial ware; and (*c*) *fair*, glazes whose texture was below commercial standards.

(iii) *Blemishes:* All blemishes, such as blisters, pinholes, crawling, and pitting, were recorded as such and are so named in the data on the glazes.

[1] C. J. Koenig, "Low-Temperature Vitreous Bodies," *Jour. Amer. Ceram. Soc.*, **25** [9] 230–36 (1942).

[2] (*a*) H. Harkort, "Method of Testing Crazing of Whiteware," *Trans. Amer. Ceram. Soc.*, **15**, 368–72 (1913).
(*b*) J. W. Mellor, "Crazing and Peeling of Glazes," *Trans. Ceram. Soc.* [England], **34**, 1–112 (1934–1935); *Ceram. Abs.*, **14** [9] 233 (1935).

(*D*) *Preparation and Firing of Glazes:* All frits were weighed out in 2000-gm. lots and smelted in fireclay crucibles in a Denver furnace. The dried frit was ground for 15 hours with a double charge of flint pebbles and screened through 80-mesh. The glazes were prepared in 1000-gm. lots and ground for 15 hours with an equal charge of pebbles. The glaze was adjusted to viscosity of 19 seconds per 200 cc. and applied to the ware by spraying. All laboratory glost firings were to cone 01 down, cone 1 standing. In the two plant trials, cone plaques placed inside the sagger with the ware showed cone 02 down, cone 01 standing or at one o'clock.

## III. Laboratory Research

The present work is a continuation of a series of twenty-five experimental glazes developed by Koenig.* Starting with standard cone 4 to 5 dinnerware glaze, he applied the following three principles to lower the maturing temperature and to adjust its properties to the syenite body:

(1) Introduction of silica in the fused or combined state.
(2) Increasing the multiplicity of the RO group: (*a*) alkaline earths BaO, BeO, and MgO replaced part of the CaO content and (*b*) $Li_2O$ replaced a portion of the alkali content.
(3) Introduction of small percentages of fluorine in the $RO_2$ group.

The successful use of fluorine in glazes is reported on in a number of recent German articles.[3]

The formulas of the standard glaze and of Koenig's glaze No. C–21, one of several excellent glazes from his series, are given as follows:

Standard cone 4–5 glaze

$$\left.\begin{matrix}0.26\ PbO\\ .12\ K_2O\\ .06\ Na_2O\\ .43CaO\\ .13ZnO\end{matrix}\right\}\ \left.\begin{matrix}0.27\ Al_2O_3\\ 0.31\ B_2O_3\end{matrix}\right\}\ \{2.6\ SiO_2$$

Glaze C–21, cone 01*

$$\left.\begin{matrix}0.26\ PbO\\ .05\ KNaO\\ .05\ Li_2O\\ .28\ CaO\\ .10\ MgO\\ .13\ SrO\\ .13\ BeO\end{matrix}\right\}\ \left.\begin{matrix}0.27Al_2O_3\\ 0.31\ B_2O_3\end{matrix}\right\}\ \{2.8SiO_2$$

* See footnote 3, p. 33.

All materials for glaze C–21 were fritted except for a portion of the CaO, $Al_2O_3$, and $SiO_2$, and the glaze used the following batch†: frit C–21 78.0, whiting 4.3, clay 12.9, flint 4.8, a total of 100%.

To complete this work started by Koenig, the following objectives were decided on: (1) to produce cone 02 to 01 glazes without the oxides of lithium or beryllium, which war restrictions have made difficult to obtain, (2) to produce a range of glazes containing varying amounts of those oxides which affect color so that the manufacturer of underglaze color ware will have the opportunity of choice, and (3) to increase the resistance to crazing of the glazes.

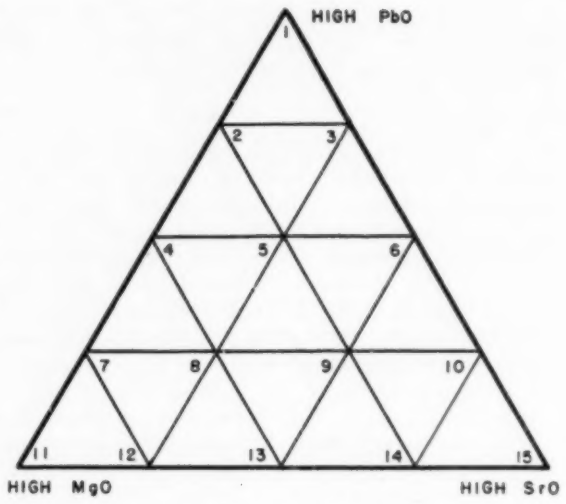


FIG. 1.—Triaxial I.

TABLE II

PERCENTAGE BATCH FORMULAS SHOWN IN TRIAXIALS I AND II (FIGS. 1 AND 2)

| | Batch formulas of frits in end members | | | | | |
|---|---|---|---|---|---|---|
| | Triaxial I | | | Triaxial II | | |
| Material | No. 1 | No. 11 | No. 15 | No. 1 | No. 16 | No. 21 |
| Red lead | 30.6 | 20.4 | 10.2 | 18.5 | 29.5 | 17.9 |
| Boric acid | 7.9 | 13.2 | 12.5 | | 8.6 | 9.0 |
| Borax | 5.8 | | | 18.4 | 3.8 | 4.0 |
| Whiting | 4.9 | 5.5 | 5.2 | 1.9 | 1.7 | |
| Dolomite | | | | 5.8 | 5.3 | 8.9 |
| Talc | 3.9 | 12.3 | 4.1 | | | 1.1 |
| $SrCO_3$ | 3.2 | 3.6 | 12.0 | | | |
| $BaCO_3$ | | | | 6.1 | 5.6 | 11.9 |
| Nepheline syenite | | 7.8 | 7.3 | | | |
| Oxford Maine feldspar | | | | 15.4 | | |
| Buckingham feldspar | | | | | 2.5 | 2.6 |
| Clay | 13.3 | 10.2 | 9.6 | 7.1 | 12.0 | 12.7 |
| Flint | 30.4 | 27.0 | 30.1 | 26.8 | 31.0 | 31.9 |
| | Batch formulas of end members | | | | | |
| Frit (see above) | 81.2 | 77.4 | 78.1 | 81.8 | 83.3 | 82.1 |
| Whiting | 3.0 | 3.6 | 3.5 | 4.2 | 3.8 | 4.1 |
| $SrCO_3$ | 2.2 | 2.7 | 2.6 | | | |
| Clay | 6.5 | 7.8 | 7.5 | 6.9 | 6.3 | 6.8 |
| Flint | 7.1 | 8.5 | 8.3 | 7.1 | 6.6 | 7.0 |

Through the use of two triaxial studies, combinations of oxides were studied which could replace the $Li_2O$ and BeO. In Fig. 1, triaxial I uses glaze C–21 as its base. The elimination of 0.13 molecule of BeO and 0.05 molecule of $Li_2O$ from this glaze leaves a vacancy of 0.18 molecule in the RO group which had to be filled. Triaxial I uses additional amounts of PbO, MgO, and SrO, respectively, in the three corners to complete the base

* C. J. Koenig, formerly with the Engineering Experiment Station, Ohio State University, and now with the armed forces.

[3] C. J. Koenig, "Nepheline Syenite in Low-Temperature Vitreous Ware," *Ohio State Univ. Eng. Expt. Station Bull.*, No. 112, 36 pp. (1942); *Ceram. Abs.*, **22** [5] 77 (1943); p. 20.

† See footnote 3, p. 33.

TABLE III

MOLECULAR FORMULAS OF EACH OF FIFTEEN POINTS ON TRIAXIAL I

| Point No. | Oxides* PbO | MgO | SrO |
|---|---|---|---|
| 1 | 0.440 | 0.100 | 0.130 |
| 2 | .395 | .145 | .130 |
| 3 | .395 | .100 | .175 |
| 4 | .350 | .190 | .130 |
| 5 | .350 | .145 | .175 |
| 6 | .350 | .100 | .220 |
| 7 | .305 | .235 | .130 |
| 8 | .305 | .190 | .175 |
| 9 | .305 | .145 | .220 |
| 10 | .305 | .100 | .265 |
| 11 | .260 | .280 | .130 |
| 12 | .260 | .235 | .175 |
| 13 | .260 | .190 | .220 |
| 14 | .260 | .145 | .265 |
| 15 | .260 | .100 | .310 |

* Other oxides were constant as follows: CaO 0.280, KNaO 0.050, $Al_2O_3$ 0.27, $B_2O_3$ 0.31, and $SiO_2$ 2.80.

group. The interior points on the triaxial thus provide the opportunity for these three oxides to combine in varying proportions.

Batch formulas for the six frits and for the glaze batches used in the end members of the two triaxials are given in Table II. The fifteen points of triaxial I are shown in Fig. 1; the complete molecular formula for each glaze contained in the triaxial is shown in Table III; and the properties of the glazes in Table IV.

All glazes were fully matured at cone 01. The most promising glazes were Nos. 4, 5, and 10, which were the most pleasing to the eye and showed good fit. The three high-lead glazes had too great a gloss resulting in a wetness which gave the glaze an unpleasant sheen. The same condition was present to a lesser extent in glazes Nos. 14 and 15, high in strontia. This fault, however, was absent from glaze No. 10, containing 0.265 molecule of strontia.

The MgO content in these glazes is important inasmuch as its low expansion is very beneficial to glaze fit. Its refractory nature sets a limit on the amount that can be safely employed without injuring gloss and surface texture. The triaxial was such that the maximum limit of MgO could be determined quite accurately. For this type of glaze, MgO can be used up to and including 0.19 molecular equivalent without in any way reducing gloss, impairing surface texture, or causing any immaturity. When it occurs in the amount of 0.28 molecule, as is the case in glaze No. 11, the glaze becomes mat and low in gloss but retains a very smooth texture. Glazes Nos. 7 and 12, which contained 0.235 molecule of MgO, were in between a bright and a mat glaze and consequently had the poorest surface texture in the triaxial.

The oxides of barium and zinc were substituted for the strontia in glaze No. 4 as glazes Nos. 4A and 4B, respectively. Zinc glaze No. 4B was the equal of the triaxial glaze in every respect and glaze No. 4A was only slightly inferior as to gloss.

Triaxial I showed that glazes could mature at cone 01 with only 0.05 molecule of sodium and potassium oxides

TABLE IV

PROPERTIES OF EACH OF FIFTEEN POINTS ON TRIAXIAL I*

| Point† No. | Gloss | Surface texture | Thermal shock (200°–25°C) Cycle | Comments |
|---|---|---|---|---|
| 1 | High | Excellent | | Wet looking |
| 2 | " | Good | Failed 5th | " " |
| 3 | " | Excellent | " 4th | " " |
| 4 | " | " | " 5th | One of best |
| 5 | " | " | " 5th | " " " |
| 6 | " | Good | " 1st | |
| 7 | Medium | Fair | | |
| 8 | High | Good | Failed 5th | |
| 9 | " | " | " 4th | |
| 10 | " | Excellent | " 3d | One of best |
| 11 | Low | Good | Passed 5 | Smooth mat |
| 12 | Medium | Fair | Failed 2d | Pinholes |
| 13 | High | Good | " 1st | " |
| 14 | " | " | Passed 5 | Wet looking |
| 15 | " | Excellent | " 5 | " " |

* Glost fire to cone 01 on a nepheline syenite body, similar to No. 11, with an absorption of 1.5%.
† Nos. 1, 4, and 7 passed the autoclave test.

TABLE V

MOLECULAR FORMULAS OF EACH OF TWENTY-ONE POINTS ON TRIAXIAL II

| Point No. | Oxides* PbO | MgO | BaO | KNaO |
|---|---|---|---|---|
| 1 | 0.260 | 0.100 | 0.100 | 0.240 |
| 2 | .298 | .100 | .100 | .202 |
| 3 | .260 | .118 | .120 | .202 |
| 4 | .336 | .100 | .100 | .164 |
| 5 | .298 | .118 | .120 | .164 |
| 6 | .260 | .136 | .140 | .164 |
| 7 | .374 | .100 | .100 | .126 |
| 8 | .336 | .118 | .120 | .126 |
| 9 | .298 | .136 | .140 | .126 |
| 10 | .260 | .154 | .160 | .126 |
| 11 | .412 | .100 | .100 | .088 |
| 12 | .374 | .118 | .120 | .088 |
| 13 | .336 | .136 | .140 | .088 |
| 14 | .298 | .154 | .160 | .088 |
| 15 | .260 | .172 | .180 | .088 |
| 16 | .450 | .100 | .100 | .050 |
| 17 | .412 | .118 | .120 | .050 |
| 18 | .374 | .136 | .140 | .050 |
| 19 | .336 | .154 | .160 | .050 |
| 20 | .298 | .172 | .180 | .050 |
| 21 | .260 | .190 | .200 | .050 |

* Other oxides were constant as follows: CaO 0.300, $Al_2O_3$ 0.27, $B_2O_3$ 0.31, and $SiO_2$ 2.8.

and the glaze could benefit by the consequent reduction in thermal expansion. Employing these two alkalis in greater quantity, however, provides the opportunity to introduce them in the glaze batch as feldspar or syenite and thus to reduce the frit content of the glaze. With this procedure in mind, triaxial II was set up with a high alkali corner, a high lead corner, and the third corner high in the oxides of magnesium and barium. Strontia was entirely absent from this triaxial since it was on high priority at the time. In order that the frit content be held constant throughout the triaxial, all alkali was put into the frit with the intention of changing to the feldspar batch component after the best combination of oxides had been determined. All of the glaze formulas except 0.14 CaO, 0.09 $Al_2O_3$, and 0.58 $SiO_2$ were fritted.

TABLE VI

PROPERTIES OF EACH ON TWENTY-ONE POINTS ON TRIAXIAL II*

| Point No. | Surface texture† | Autoclave | Thermal shock (200°–25°C.) cycle C | | Comments |
|---|---|---|---|---|---|
| 1 | Good | | Failed | 1st | |
| 2 | " | | " | " | |
| 3 | " | Passed | | | |
| 4 | " | | Failed | 1st | |
| 5 | " | | " | " | |
| 6 | " | | " | " | |
| 7 | Excellent | | " | " | One of best |
| 8 | " | Passed | | | " " " |
| 9 | " | | Failed | 2nd | " " " |
| 10 | Good | | " | 5th | |
| 12 | " | Passed | | | |
| 13 | " | | Failed | 1st‡ | |
| 14 | Excellent | | " | 4th‡ | |
| 15 | Good | | " | 1st | |
| 18 | " | | " | 1st‡ | |
| 19 | " | | " | 1st‡ | |
| 20 | " | | Passed | 5 | |
| 21 | " | | Failed | 5th‡ | |

* Glost fire to cone 01 on a vitreous body which took no ink stain; glazes Nos. 11, 16, and 17 not tried because of high lead content.

† All specimens had high gloss.

‡ Failed by dunt in form of crack penetrating both body and glaze.

The twenty-one point triaxial and the molecular formulas of each glaze contained therein are shown in Fig. 2 and Table V. Table VI contains data on the physical and visual properties of the glazes.

All glazes in triaxial II were completely mature at cone 01. Glazes Nos. 15, 20, and 21, high in BaO and MgO, showed a certain rippled surface from the spray application indicating a viscosity not apparent in the other glazes. The best-looking glazes were Nos. 9 and 14 containing 0.298 PbO and 0.126 and 0.088 KNaO, respectively. They were equal in gloss and surface texture to any of the higher alkaline glazes, all of which had a very good appearance.

When the glazes were subjected to the thermal-shock test for crazing, the most significant division in the triaxial was made apparent. Glazes Nos. 1 to 7, inclusive, failed in the first cycle of a 200° to 25°C. test. The crazing was general all over the plates and was not restricted to a small crack over the edge or foot rim, which was usually the case with failures in triaxial I and with the lower alkali glazes of triaxial II.

Glazes Nos. 3, 8, and 12, which varied only in the lead and alkali content, were subjected to the autoclave test which all passed. Sixteen months have passed at the time of writing and an examination of the three plates shows that no crazing has occurred during the period.

## IV. Plant Trials

Four glazes which had shown the greatest promise in the laboratory were selected to be tried in a commercial cone 02 kiln. These glazes were No. 4A, which was point No. 4 in triaxial I with barium oxide substituted

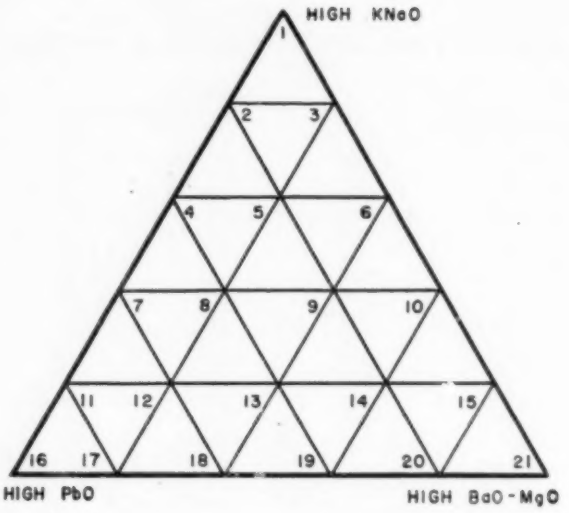


FIG. 2.—Triaxial II.

for the 0.13 molecular equivalent of strontia, glaze No. 4B, which was the same glaze with zinc oxide in place of the strontia; glaze No. 5, which was point No. 5, in triaxial I with the strontia included; and glaze W, which was similar in molecular composition to the area between points Nos. 9 and 14 in triaxial II but with a lower frit content and the inclusion of feldspar in the batch.

Frits for these four glazes were smelted in quantity* and were designated as PL–482, PL–486, PL–487, and PL–488, respectively, for the four frits. Two additional glazes were added to the list in the second plant trial. These were built around two frits made by Pemco, P–384 and P–389, which contain seven oxides in the RO group including lithia. Glaze P–389 uses the batch additions recommended by Pemco for the frit. Glaze 384–B was developed by the writer using the lithia frit but introducing strontium carbonate instead of zinc oxide as a mill addition and increasing the MgO content.

The glazes were ground 16 hours at the plant and were applied to the plates, rings, and bars by dipping. The 5-in. plates used in the first plant trial had an absorption of 2.3%; the plates in the second plant trial were fired to an absorption of 0.5%; the rings had 0.0% absorption; and the bars 0.13% absorption.

All ware was glost fired to cone 02 in the tunnel kiln at the plant.

Molecular and batch formulas for the glazes and frits used in the plant trial are presented in Tables VII through X. Table XI shows the percentage composition of the various glazes according to the weight of the oxides and also the total percentage of the "soft" oxides occurring in the glazes. For the purpose of comparison, the same figures are given for the standard cone 5 dinnerware glaze. Three of the low-temperature glazes contain from 0.01 to 0.86% more of these "soft" oxides;

* These glazes were smelted at the plant of Pemco Corp., Baltimore, Md.

TABLE VII
MOLECULAR FORMULAS OF SIX GLAZES IN PLANT TRIALS

| Glaze | Oxide | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $Li_2O$ | $K_2O$ | $Na_2O$ | PbO | CaO | MgO | BaO | SrO | ZnO | $Al_2O_3$ | $B_2O_3$ | $SiO_2$ |
| 4A | | 0.024 | 0.026 | 0.350 | 0.280 | 0.190 | 0.130 | | | 0.27 | 0.310 | 2.8 |
| 4B | | .024 | .026 | .350 | .280 | .190 | | | 0.130 | .27 | .310 | 2.8 |
| 5 | | .024 | .026 | .350 | .280 | .145 | | 0.175 | | .27 | .310 | 2.8 |
| W | | .064 | .036 | .300 | .330 | .130 | 0.140 | | | .27 | .310 | 2.8 |
| 384–B | 0.041 | .042 | .017 | .265 | .202 | .202 | .031 | 0.200 | | .27 | .310 | 2.8 |
| P–389 | .041 | .040 | .120 | .260 | .278 | .100 | .030 | | 0.131 | .27 | .304 | 2.9 |

TABLE VIII
BATCH FORMULAS OF SIX GLAZES IN PLANT TRIALS

| Material | Glaze (%) | | | | | |
|---|---|---|---|---|---|---|
| | 4A | 4B | 5 | W | 384–B | P–389 |
| Frit PL–482 | 81.20 | | | | | |
| Frit PL–486 | | 80.69 | | | | |
| Frit PL–487 | | | 81.02 | | | |
| Frit PL–488 | | | | 67.34 | | |
| Frit P–384 | | | | | 66.70 | |
| Frit P–389 | | | | | | 58.61 |
| Nepheline syenite | | | | | | 20.25 |
| Buckingham feldspar | | | | 12.93 | 8.01 | |
| Whiting | 3.42 | 3.51 | 3.45 | 4.04 | | 5.26 |
| Dolomite | | | | | 5.25 | |
| Strontium carbonate | | | | | 8.46 | |
| Zinc oxide | | | | | | 3.15 |
| Clay | 7.35 | 7.55 | 7.42 | 6.69 | 10.83 | 2.88 |
| Flint | 8.03 | 8.25 | 8.11 | 9.00 | 0.75 | 9.86 |

TABLE IX
MOLECULAR FORMULAS OF SIX FRITS USED IN PLANT TRIALS

| Frit | Oxide | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | $Li_2O$ | $K_2O$ | $Na_2O$ | PbO | CaO | MgO | BaO | SrO | ZnO | $Al_2O_3$ | $B_2O_3$ | $SiO_2$ |
| PL–482 | | 0.027 | 0.030 | 0.3975 | 0.182 | 0.216 | 0.1475 | | | 0.193 | 0.3525 | 2.42 |
| PL–486 | | .027 | .030 | .3975 | .182 | .216 | | | 0.1475 | .193 | .3525 | 2.42 |
| PL–487 | | .027 | .030 | .3975 | .182 | .165 | | 0.1985 | | .193 | .3525 | 2.42 |
| PL–488 | | | .026 | .3845 | .243 | .167 | .1795 | | | .117 | .3974 | 2.08 |
| P–384 | 0.075 | .004 | .013 | .4810 | .186 | .185 | .0560 | | | .123 | .5630 | 3.93 |
| P–389 | .075 | .004 | .013 | .4810 | .186 | .185 | .0560 | | | .123 | .5630 | 2.94 |

the other three contain from 1.57 to 3.67% less. The increase in PbO in some of these glazes is thus counteracted by reduced alkali and higher silica so that the total content of "soft" materials is kept fairly constant.

Flux-block tests of glazes 4A, 4B, 5, and W, fired to cone 08, are shown in Fig. 3. A comparison is made between two glazes of the same molecular formula with one being compounded out of two frits and the other with one. The formula is that of glaze No. 4 in triaxial I. The left-hand flux block contains the glaze as it was originally mixed for the triaxial using a blend of the high-lead and the high-magnesium frits. The right-hand block contains the identical formula with both frits smelted as one, resulting in increased fluidity.

Test data from the plant trials are contained in Tables XII through XV.

The percentage of solubility for four of the test frits was as follows: PL–482 0.6, PL–486 0.4, PL–487 0.7 and PL–488 3.9.

## (1) Appearance of Glazes

All glazes had good gloss and texture and were free from any blemishes. In the first plant trial, glazes 5 and W had the best all-around appearance. Glaze 4B showed a slightly wavy surface on some plates, and glaze 4A had a little lower gloss than the rest. When fired on the nearly vitreous ware of the second plant trial, glaze 5 had the highest gloss with 4B and W close behind it. The rippled surface of glaze 4B seemed to have smoothed out on the vitreous ware. Glaze 4A had still lower gloss and took on the appearance of a very smooth, soft, vellum mat glaze the second time it was

TABLE X
BATCH FORMULAS OF FOUR FRITS USED IN PLANT TRIALS

| Material | Frit (%) | | | |
|---|---|---|---|---|
| | PL–482 | PL–486 | PL–487 | PL–488 |
| Red lead | 24.20 | 25.40 | 24.35 | 24.71 |
| Boric acid | 10.10 | 10.60 | 10.22 | 12.10 |
| Borax | 2.30 | 2.40 | 2.33 | 2.76 |
| Whiting | | | 0.46 | 2.16 |
| Dolomite | 8.90 | 9.40 | 8.14 | 8.64 |
| Talc | 1.20 | 1.20 | | |
| $SrCO_3$ | | | 7.90 | |
| $BaCO_3$ | 7.80 | | | 9.96 |
| Zinc oxide | | 3.30 | | |
| Buckingham feldspar | 5.10 | 5.30 | 5.13 | |
| Clay | 10.70 | 11.20 | 10.79 | 8.54 |
| Flint | 29.70 | 31.20 | 30.68 | 31.13 |

TABLE XI

PERCENTAGE COMPOSITION OF OXIDES IN SIX CONE 01 GLAZES AND STANDARD CONE 5 GLAZE

| Oxide | Glaze (%) 4A | 4B | 5 | W | 384-B | P-389 | Standard |
|---|---|---|---|---|---|---|---|
| PbO | 22.80 | 23.44 | 23.03 | 19.82 | 18.04 | 17.67 | 18.53 |
| $K_2O$ | 0.66 | 0.68 | 0.66 | 1.78 | 1.23 | 1.17 | 3.60 |
| $Na_2O$ | 0.47 | 0.48 | 0.48 | 0.66 | 0.32 | 2.27 | 1.19 |
| $Li_2O$ | | | | | 0.38 | 0.37 | |
| CaO | 4.58 | 4.71 | 4.63 | 5.48 | 3.45 | 4.74 | 7.70 |
| MgO | 2.22 | 2.28 | 1.71 | 1.54 | 2.47 | 1.22 | |
| BaO | 5.81 | | | 6.35 | 1.45 | 1.10 | |
| SrO | | | 5.37 | | 6.35 | | |
| ZnO | | 3.17 | | | | 3.23 | 3.37 |
| $Al_2O_3$ | 8.04 | 8.27 | 8.13 | 8.16 | 8.41 | 8.40 | 8.80 |
| $B_2O_3$ | 6.34 | 6.52 | 6.41 | 6.43 | 6.62 | 6.49 | 6.94 |
| $SiO_2$ | 49.08 | 50.45 | 49.58 | 49.78 | 51.28 | 53.04 | 49.87 |
| Total PbO, $K_2O$, $Na_2O$, $Li_2O$, $B_2O_3$ | 30.27 | 31.12 | 30.58 | 28.69 | 26.59 | 27.97 | 30.26 |

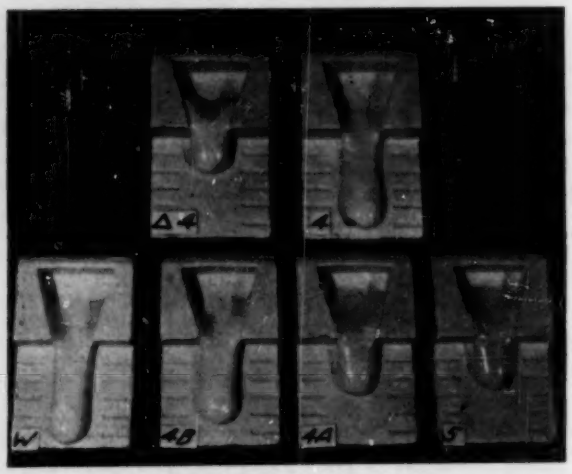


FIG. 3.—Flux-block tests of cones 02 to 01 glazes; all fired at cone 08.

fired. The firings indicated that any of these three glazes, 4B, 5, and W, could be counted on to give a bright smooth glaze. The two lithium glazes had exceptionally high gloss of a richness which only lithium can produce. Glaze P-389, using the frit and batch recommended by Pemco, combined a very smooth texture with the high gloss. Glaze 384-B showed some pinholed areas owing to its higher MgO content and increased viscosity; although some plates were very good, this glaze cannot be recommended to give consistent results.

### (2) Analysis of Test Data

None of the glazed ware showed any crazing or dunting unless subjected to the various tests. At the time of writing, approximately six months have elapsed since the plant trials, and an examination of samples shows that all plates not crazed in a test have had no tendencies to craze by themselves. These periodic examinations will be continued in the future.

TABLE XII

MODULUS OF RUPTURE AND GLAZED-RING TEST RESULTS

| Glaze | Modulus of rupture test (lb./sq. in.) Min. | Max. | Avg. |
|---|---|---|---|
| 4A | 7410 | 10,850 | 9648 |
| 4B | 7140 | 10,520 | 9520 |
| 5 | 8320 | 10,230 | 9352 |
| W | 8100 | 9,270 | 8813 |
| Unglazed | 6730 | 9,450 | 8746 |
| | Glazed-ring test (mm. of compression) | | |
| 4A | −0.0328 | −0.0656 | −0.0472 |
| 4B | − .0246 | − .0574 | − .0410 |
| 5 | − .0246 | − .0615 | − .0441 |
| W | − .0246 | − .0492 | − .0369 |

TABLE XIII

AUTOCLAVE TEST RESULTS

| Glaze | No. of failures each cycle 1st | 2d | 3d | 4th | 5th | No. passed | Avg. cycle passed |
|---|---|---|---|---|---|---|---|
| First plant trial (8 plates of each glaze tested, absorption 2.3%) | | | | | | | |
| 4A | 5 | 1 | 1 | 0 | 1 | 0 | 0.9 |
| 4B | 0 | 1 | 1 | 1 | 0 | 5 | 3.9 |
| 5 | 4 | 1 | 0 | 0 | 1 | 2 | 1.9 |
| W | 5 | 3 | | | | 0 | 0.4 |
| Second plant trial (4 plates of each glaze tested, absorption 0.5%) | | | | | | | |
| 4A | 0 | 0 | 0 | 0 | 0 | 4 | 5.0 |
| 4B | 0 | 0 | 0 | 0 | 0 | 4 | 5.0 |
| 5 | 0 | 0 | 0 | 0 | 0 | 4 | 5.0 |
| W | 0 | 0 | 0 | 0 | 1 | 3 | 4.8 |
| 384-B | 0 | 0 | 0 | 0 | 0 | 4 | 5.0 |
| P-389 | 1 | 0 | 0 | 1 | 0 | 2 | 3.2 |

The first important fact shown by the test data is that glazes 4A, 4B, and 5, in which the total alkali was limited to 0.05 molecule, showed the best resistance to crazing. They produced the greatest strength in modulus of rupture, showed the highest compression in the ring test, had the fewest failures in autoclave and thermal-shock tests, had the highest impact values, and also showed the lowest frit solubility.

The one significant test in which these glazes were surpassed was in scratch hardness where the smallest

TABLE XIV

THERMAL-SHOCK TEST RESULTS

| Glaze | 175°–25°C. 1st | 2d | 3d | 4th | 5th | 200°–25°C. 6th | 7th | 8th | 9th | 10th | No. passed | Avg. cycle passed |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **First plant trial (9 plates of each glaze tested, absorption 2.3%)** | | | | | | | | | | | | |
| Failures by body dunt | | | | | | | | | | | | |
| 4A | 1 | 1 | 1 | 1 | 0 | 2 | 3 | | | | 0 | 3.8 |
| 4B | 1 | 2 | 2 | 0 | 1 | 2 | 0 | 0 | 0 | 0 | 1 | 3.3 |
| 5 | 1 | 1 | 0 | 0 | 0 | 3 | 4 | | | | 0 | 4.4 |
| W | 2 | 1 | 0 | 1 | 0 | 3 | 1 | 1 | | | 0 | 3.6 |
| Failures by crazing | | | | | | | | | | | | |
| 4A | 0 | 0 | 0 | 0 | 0 | 1 | 3 | 1 | 4 | | 0 | 6.9 |
| 4B | 0 | 0 | 0 | 0 | 0 | 1 | 1 | 1 | 3 | 1 | 2 | 7.9 |
| 5 | 0 | 0 | 0 | 0 | 0 | 2 | 4 | 0 | 3 | | 0 | 6.4 |
| W | 0 | 0 | 0 | 0 | 0 | 5 | 3 | 1 | | | 0 | 5.6 |
| **Second plant trial (three plates of each glaze tested, absorption 0.5%)* (Failures by crazing at 175°–25°C.)** | | | | | | | | | | | | |
| | 1st | 2d | 3d | 4th | 5th | 6th | 7th | 8th | 9th | 10th | | |
| 4A | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 3 | 10.0 |
| 4B | 0 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 2 | 7.7 |
| 5 | 0 | 0 | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 2 | 7.3 |
| W | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 2 | 6.7 |
| 384–B | 3 | | | | | | | | | | 0 | 0 |
| P–389 | 3 | | | | | | | | | | 0 | 0 |

* Three plates each of glazes 4A, 4B, 5, and W were subjected to one cycle of a thermal shock from 200° to 25°C.; all showed craze marks and body dunts at the end of the first cycle.

scratch (2.8) occurred on glaze P–389. This glaze contained 2.9 $SiO_2$ as compared with the 2.8 $SiO_2$ in the other glazes, and the increased silica showed its benefit in this test. Glazes 4A and 5 had the same scratch-hardness values (2.9) as the commercial hotel china glaze, and although glazes 4B and W (3.0 scratch hardness) showed an average increase in the width of the scratch of 0.1 micron, it was 0.2 micron less than the scratch (3.2) on a commercial semivitreous, cone 6 glaze; glaze 384–B had a width of 3.1 micron.

In the modulus of rupture test, all four glazes increased the strength of the body from an average of 67 p.s.i. for glaze W to 952 p.s.i. for glaze 4A. The three lowest alkali glazes showed an average compression of between 0.04 and 0.05 mm. existing between themselves and the body. The autoclave test on ware with a 2.3% absorption showed zinc glaze 4B to have the best resistance to moisture-expansion crazing. When the absorption of the body was reduced to 0.5%, there was no failure with the three low alkali glazes in five cycles of the autoclave. The thermal-shock tests showed glazes 4A and 4B to stand up the longest before crazing. The 175° to 25°C. shock on the ware with 0.5% absorption showed no body failures and few crazing failures. Glazes 4A, 4B, 5, and W passed an average of over five cycles of this test. When the top temperature was increased to 200°C., it was very close to the cristobalite inversion point and the increased strains resulted in failure by body cracks occurring before any crazing. Plates with glaze 4B showed the poorest resistance to the body dunt type of failure but the best resistance to crazing. On the ware with 2.3% absorption, the body type of failure occurred in the test at 175° to 25°C., and no crazing took place until the top temperature was increased to 200°C.

The impact and chipping tests were both influenced by the physical proportions of the ware. The 5-in. test plates were jiggered in semivitreous-ware plants on semivitreous molds with the cutting knife adjusted to produce a fired plate with a thickness of 0.13 in. The edges of the plates in the first plant trial were plain but those used in the second plant trial had a relief decoration at the edge, resulting in a thinner cross section at the point of contact with the chipping hammer and consequently lower chipping values. On the whole, chipping tests made on any ware other than rolled-edge hotel ware have little significance for comparative glaze values. The impact test, however, will show glaze compression, which was the case in the present testing where the three low-alkali glazes had the highest impact values.

TABLE XV

IMPACT AND CHIPPING TEST RESULTS

| Glaze | First plant trial Min. | Max. | Avg. | Second plant trial Min. | Max. | Avg. |
|---|---|---|---|---|---|---|
| Impact test, avg. plate thickness, 0.13 in. (lb./in. thickness) | | | | | | |
| 4A | 3.4 | 7.8 | 5.3 | 6.0 | 6.1 | 6.1 |
| 4B | 4.4 | 6.3 | 5.1 | 4.5 | 6.1 | 5.3 |
| 5 | 4.6 | 6.5 | 6.0 | 4.1 | 4.4 | 4.3 |
| W | 4.1 | 5.7 | 4.9 | 3.7 | 4.8 | 4.4 |
| 384–B | | | | 2.8 | 3.1 | 3.0 |
| P–389 | | | | 3.3 | 4.4 | 3.7 |
| Chipping test (ft.-lb.) | | | | | | |
| 4A | 0.088 | 0.129 | 0.094 | 0.058 | 0.061 | 0.059 |
| 4B | .084 | .093 | .088 | .049 | .075 | .062 |
| 5 | .101 | .136 | .117 | .048 | .049 | .048 |
| W | .087 | .097 | .093 | .051 | .076 | .063 |
| 384-B | | | | .056 | .071 | .063 |
| P–389 | | | | .050 | .054 | .052 |

In general, the two glazes which showed up to best advantage both as to appearance and physical properties are the zinc glaze, 4B, and the strontium glaze, 5.

## V. Summary and Conclusions

(1) Glazes maturing at cones 02 to 01 and fitting a vitreous nepheline syenite body biscuited at cones 4 to 5 can be produced.

(2) To insure the best glaze fit, the total alkali content ($K_2O$, $Na_2O$, and $Li_2O$) should not exceed 0.05 molecule and the MgO content should not be below 0.15 molecule.

(3) To produce adequate fusibility, the PbO content should be between 0.30 and 0.35 molecule.

(4) One or more of the following oxides should be used with CaO to fill the remainder of the RO group, ZnO, SrO, and BaO. The use of barium and strontium oxides makes possible the production of zincless glazes where that oxide is detrimental to underglaze colors.

(5) The total percentage by weight of those oxides producing "soft" glazes need not, and should not, be above the 30.3% found in the standard cone 5 dinner-ware glaze; these oxides include PbO, $K_2O$, $Na_2O$, $Li_2O$, and $B_2O_3$.

(6) The $Al_2O_3$ and $B_2O_3$ content of the standard cone 5 glaze need not be altered for the low-temperature glazes.

(7) The property of the nepheline syenite bodies to take up silica makes possible an increase in the $SiO_2$ content of the standard glaze from 2.6 to 2.8 molecule, resulting in better hardness and greater resistance to crazing and acid attack. Presmelting most of the silica in the frit insures the complete maturity of the glaze at cones 02 to 01. Such frits have been successfully smelted by a commercial manufacturer.

**Acknowledgment**

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# ABRASION RESISTANCE OF PORCELAIN ENAMELS*

By Clark Hutchison

Abstract

The abrasion resistances of enamels, differing in composition and properties, were determined by the test for resistance of porcelain enamels to surface abrasion (a standard of the Porcelain Enamel Institute, March, 1942).

Four to five classes of abrasion resistance were obtained, but for the most part, the results fell within relatively narrow limits. Differences in abrasion index, in general, were not distinguishable by visual inspection. No one class or kind of enamel was superior. While changes in abrasion index were affected by changes in frit formula, it seems that many compositions will give comparable results.

## I. Introduction

Porcelain enamel is often described as the "lifetime finish." Inasmuch as many porcelain enameled products are subjected to surface abrasion, good abrasion resistance is important from the standpoint of durability.

The tests herein described were conducted primarily to determine the difference in surface-abrasion resistance likely to be encountered with various compositions and types of porcelain enamels and to note whether such inherent properties as acid resistance, refractoriness, or specular gloss might be indicative of abrasion resistance.

Published results,[1] based on the P.E.I. surface-abrasion test,[2] have shown significant differences between various enamels, and acid-resisting enamels are reported as generally giving best results.

## II. Procedure

The abrasion test used[2] consists essentially of (1) reading specular gloss of the test specimens (11 cm. square), (2) subjecting them to a controlled abrasive treatment, and (3) again reading specular gloss. The percentage of gloss retained is the abrasion index or the measure of abrasion resistance.

The time necessary to reduce the gloss of standard glass plates by 50% determines the length of treatment (9.4 minutes total for these tests). Standard glass plates are also run with test specimens as a check on the accuracy of operations.

* Presented at the Forty-Sixth Annual Meeting, The American Ceramic Society, Pittsburgh, Pa., April 5, 1942 (Enamel Division). Received April 11, 1942.

[1] (*a*) W. N. Harrison, "Tentative Tests for Abrasion Resistance," *Proc. Third Annual Forum, Porcelain Enamel Institute, Oct. 13, 1938*, pp. 32–37; *Ceram. Abs.*, **18** [6] 150 (1939).

(*b*) P. C. McCollom and R. R. Danielson, "Effect of Firing Treatment on Physical Properties of Dry-Process Enamels," *Jour. Amer. Ceram. Soc.*, **25** [Jan. 15, No. 2] 48–50 (1942).

(*c*) Clark Hutchison, "Effect of Various Factors on Abrasive Resistance of Porcelain Enamels," *Bull. Amer. Ceram. Soc.*, **18** [6] 202–205 (1939).

[2] Test for Resistance of Porcelain Enamels to Surface Abrasion. Standard of the Porcelain Enamel Institute, March, 1942.

TABLE I

ABRASION INDICES OF COMMERCIAL ENAMELS

| Enamel No. | Type of coat | Firing temp. (°F.) | Acid resistance | Initial specular gloss (%) | Abrasion index (%) | % confidence between | | | (%) |
|---|---|---|---|---|---|---|---|---|---|
| 1 | White* | 1500 | D | 5.44 | 71.2 ± 2.0 | 1 | and | 2 | 99.8 |
| 2 | Blue A.R.† | 1560 | A | 5.43 | 66.7 ± 0.5 | 2 | " | 5 | 0.0 |
| 3 | Blue† | 1560 | D | 5.15 | 66.6 ± 1.2 | 3 | " | 6A | 99.8 |
| 4 | Tan* | 1500 | C | 5.24 | 66.6 ± 1.1 | | | | |
| 5 | White A.R.* | 1500 | AA | 6.11 | 66.6 ± 0.8 | 5 | " | 6A | 99.9 |
| 6A | Green A.R.* | 1500 | A | 5.85 | 63.4 ± 1.0 | 6A | " | 9 | 89 |
| 6B | Black A.R.* | 1500 | B-A | 5.65 | 63.3 ± 1.5 | 6A | " | 10 | 98 |
| 6C | Tan A.R.* | 1500 | B-A | 5.61 | 62.8 ± 1.1 | 6B | " | 10 | 94 |
| 7 | White* | 1450 | D | 5.81 | 62.8 ± 1.5 | 6C | " | 10 | 91 |
| 8 | Black A.R.* | 1500 | A | 5.86 | 62.2 ± 1.0 | 6 | " | 6C | 97 |
| 9 | White* | 1540 | D | 6.09 | 62.1 ± 1.4 | 7 | " | 6 | 92 |
| 10 | Brown† | 1530 | D | 5.51 | 61.7 ± 0.7 | 8 | " | 6 | 92 |
| 6 | Clear A.R.* | 1500‡ | A | 5.67 | 60.7 ± 1.4 | 10 | " | 6D | 97 |
| 6D | Yellow A.R.* | 1500‡ | AA | 6.00 | 59.5 ± 1.6 | 6 | " | 6E | 98 |
| 6E | Red A.R.* | 1500 | A | 5.86 | 58.2 ± 1.3 | | | | |
| 11 | Brown† | 1450 | D | 5.72 | 54.9 ± 1.3 | 6E | " | 11 | 99.3 |

* Cover coat. † Ground coat. ‡ 2 Firings.

TABLE II

EFFECT OF DECREASING $SiO_2$ ON ABRASION INDEX

| Enamel No. | $SiO_2$ removed (%) | Acid resistance | Initial specular gloss (%) | Abrasion index (%) | % confidence between | | | (%) |
|---|---|---|---|---|---|---|---|---|
| 5 | 0.0 | AA | 6.10 | 68.3 ± 1.0 | 5 | and | 5A | 91 |
| 5A | 1.0 | AA | 6.09 | 67.1 ± 1.1 | 5 | " | 5E | 96 |
| | | | | | 5A | " | 5E | 85 |
| 5E | 5.0 | AA | 6.22 | 65.2 ± 2.6 | 5A | " | 5F | 93 |
| 5F | 6.0 | A | 6.17 | 65.5 ± 1.4 | 5F | " | 5J | 96 |
| | | | | | 5E | " | 5J | 93 |
| 5J | 10.0 | A | 6.33 | 62.9 ± 0.5 | 5J | " | 5M | 91 |
| | | | | | 5J | " | 5O | 99 |
| 5M | 13.0 | B | 6.42 | 61.6 ± 1.5 | 5M | " | 5O | 95 |
| 5O | 15.0 | B | 6.38 | 61.6 ± 0.7 | | | | |
| 5Q | 17.0 | C | 6.38 | 59.9 ± 0.8 | 5Q | " | 5R | 99 |
| 5R | 18.0 | C | 6.55 | 57.3 ± 0.6 | 5R | " | 5T | 0 |
| 5S | 19.0 | D | 6.41 | 57.1 ± 0.6 | | | | |
| 5T | 20.0 | D | 6.37 | 57.1 ± 0.7 | | | | |

The average of the abrasion indices for six test specimens constitutes one sample or one determination.

Acid resistance was determined according to the commercial P.E.I. acid-resistance test of porcelain enamels.[3] Briefly, the method consists of placing a few drops of citric acid (100 gm. per liter of water) on the test surface and covering with a 1-in. inverted watch glass. After 15 minutes, the acid is rinsed off and the specimen is dried and graded.

Class AA acid resistance shows no stain and passes the dry-rub pencil-mark test; class A shows a visible stain and passes the wet-rub and the blurring-highlight tests; class B fails the wet-rub test but passes the blurring-highlight test; class C fails the blurring-highlight test but passses the disappearing-highlight test; and class D fails the disappearing-highlight test.

## III. Data

In Table I are listed the test results for sixteen commercial enamels selected as representing a rather wide range of compositions and properties.

The abbreviation A.R. is used for acid resistance. The initial gloss given is the average of all readings on all plates. The abrasion index is the average (corrected by formula when necessary) of six specimens. The statistical error is given and the percentage of confidence between results is also noted.

Ground coats (Table I) were applied at 21 gm. per sq. ft.; cover coats were applied at 45 gm. per sq. ft.; and all samples were sprayed. Enamels Nos. 6, 6A, 6B, 6C, 6D, and 6E were made from the same frit, the color of the oxide being the only difference. Enamels Nos. 6D and 6E were fired a second time to remove a scum which occurred after the first fire.

To get some idea of the effect of composition changes on abrasion resistance, enamel No. 5 in Table I was selected as the base frit, and the $SiO_2$ content was progressively decreased. The effects on acid resistance, specular gloss, and abrasion resistance are noted in Table II. The application weight for these enamels was 25 gm. per sq. ft. over a fired cover coat applied at 30 gm. per sq. ft. Milling and firing were identical for each enamel.

In an effort to evaluate differences in results, visual ratings of abrasion resistance were made by two observers and compared with test results as shown in Table

[3] Test for Acid Resistance of Porcelain Enamel: I, Flatware. Standard of the Porcelain Enamel Institute, 1940; *Ceram. Abs.*, **19** [7] 158 (1940).

TABLE II

VISUAL RATINGS AS COMPARED TO TEST RESULTS

| A | | | | C | | | |
|---|---|---|---|---|---|---|---|
| Enamel No. | Abrasion index (%) | Visual rating No. 1 | Visual rating No. 2 | Enamel No. | Abrasion index (%) | Visual rating No. 1 | Visual rating No. 2 |
| 1 | 71.2 ± 2.0 | 1 | 1 | 5 | 66.6 ± 0.8 | 1 | 1 |
| 5 | 66.6 ± 0.8 | | | 1 | 71.2 ± 2.0 | 2 | |
| 7 | 62.8 ± 1.5 | | | 9 | 62.1 ± 1.4 | 3 | |
| 9 | 62.1 ± 1.4 | | | 7 | 62.8 ± 1.5 | 4 | |
| 2 | 66.7 ± 0.5 | 2 | 5 | 6D | 59.5 ± 1.6 | 5 | 2 |
| 4 | 66.6 ± 1.1 | 3 | 2 | 6 | 60.7 ± 1.4 | 6 | |
| 6C | 62.8 ± 1.1 | 4 | 3 | 2 | 66.7 ± 0.5 | 7 | 5 |
| 3 | 66.6 ± 1.1 | 5 | 4 | 3 | 66.6 ± 1.1 | 8 | 4 |
| 10 | 61.7 ± 0.7 | 6 | 6 | 6A | 63.4 ± 1.0 | 9 | 3 |
| 11 | 54.9 ± 1.3 | 7 | 7 | 6C | 62.8 ± 1.1 | 10 | |
| 8 | 62.2 ± 1.0 | 8 | 8 | 6E | 58.2 ± 1.3 | 11 | |
| | | | | 4 | 66.6 ± 1.1 | 12 | |
| B | | | | | | | |
| 6 | 60.7 ± 1.4 | 1 | 2 | 8 | 62.2 ± 1.0 | 13 | 7 |
| 6D | 59.5 ± 1.6 | 2 | 1 | 6B | 63.3 ± 1.5 | 14 | |
| 6A | 63.4 ± 1.0 | 3 | 3 | 11 | 54.9 ± 1.3 | 15 | 6 |
| 6C | 62.8 ± 1.1 | 4 | | 10 | 61.7 ± 0.7 | 16 | |
| 6E | 58.2 ± 1.3 | 5 | | | | | |
| 6B | 63.3 ± 1.5 | 6 | 4 | | | | |

III. The visual ratings were made according to dulling or loss of "eye appeal" and ratings A and B were made first. Rating C was made about three weeks later. In making these ratings, only one specimen (picked at random) from each enamel was used.

## IV. Discussion of Results

### (1) Accuracy

Abrasion indices of standard glass plates run with test specimens varied from 50.0 to 51.5 with standard deviations of 0.23 to 0.66, showing very good uniformity of treatment.

### (2) Results with Commercial Enamels

When results are grouped according to significant differences, there are four to five classes of abrasion resistance. Despite the different compositions and properties represented, however, the majority of results fall in a rather narrow range. While there is a difference of 16.3% in residual gloss between the lowest and highest results, there is only 8.5% difference among the others.

No particular type of enamel stands out as being superior in abrasion resistance. Whites are no better than other colors; cover coats as a group are no better than ground coats; and there seems to be no relation between acid resistance or "hardness" (as judged by maturing temperature) and abrasion resistance.

Higher abrasion indices, in general, were obtained with those enamels lowest in specular gloss, although no strict relationship was shown.

In addition to those listed, two full mat enamels were given the abrasion treatment. Although specular gloss measurements could not be made, there was practically no visible evidence of abrasion.

Results with the different colors made from the No. 6 enamel indicate that color oxides caused small differences in abrasion resistance, since enamels Nos. 6A, 6B, and 6C gave higher results than enamels Nos. 6, 6D, and 6E. The lower abrasion indices obtained from enamels Nos. 6D and 6E, however, may have been due to refiring.

### (3) Effect of Decreasing $SiO_2$

The results in Table II show a progressive decline in abrasion index with decreasing $SiO_2$ content. Abrasion index also decreased directly with acid resistance, but this is probably a coincidence rather than a correlation, particularly in view of results listed in Table I.

There was a trend toward increasing abrasion resistance with decreasing specular gloss, although again no strict relationship is shown.

### (4) Visual Ratings of Abrasion Resistance

As shown in Table III, visual ratings of abrasion resistance made by two observers are not in complete agreement (in fact, there are discrepancies in ratings made by the same observer at different times), but there is even less agreement with test results. Whites were unanimously picked as showing the least effects of abrasion, while the dark colors, browns and blacks, were picked as the worst. Lighter or more brilliant colors were given intermediate ratings. No attempt was made to rate the enamels visually in Table II as there was no impression of distinct difference.

Failure to visualize differences in test results does not discredit the test. It would seem, however, that large differences in abrasion resistance should be apparent to the eye. Either the differences obtained were not large or residual specular gloss is a minor factor in the visual conception of damage due to surface abrasion.

## V. Summary

Four to five grades of abrasion resistance were obtained from tests on sixteen commercial enamels made from eleven frits. Lack of test data correlated with experience, however, makes it difficult to judge the

importance of these differences in results. A rather wide difference in abrasion resistance is indicated between the best and poorest enamels, but the results, for the most part, seem to fall within a relatively narrow range.

Differences in abrasion index, in general, were not distinguishable by visual examination.

The results showed no one type or class of enamels as being superior in abrasion resistance. Although variation in formula affected abrasion resistance, apparently comparable results can be obtained with many compositions.

None of the known enamel properties offered any basis for predicting relative abrasion resistance with the possible exception of specular gloss. While no strict relationship was shown with a given group of enamels, the highest abrasion indices were obtained with those lowest in specular gloss.

If loss of specular gloss is accepted as the proper measure of abrasion resistance, the test used would seem to answer the requirements for uniformity and reproducibility.

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# EFFECT OF FLUORINE AND PHOSPHORUS PENTOXIDE ON PROPERTIES OF SODA-DOLOMITE LIME-SILICA GLASS*

CONTRIBUTION OF OWENS-ILLINOIS GLASS COMPANY GENERAL RESEARCH LABORATORY†

ABSTRACT

The effect of the substitution of fluorine for silica on the viscosity, liquidus temperature, and chemical durability of a typical soda-lime glass is shown. The effects of substituting $P_2O_5$ for (*a*) silica and (*b*) dibasic oxides in two typical soda-lime glasses are also described.

## I. Introduction

In the latter part of 1941, regular deliveries of dense soda ash to the large consumers of the material were disturbed. This situation led to an investigation of glass compositions and materials that could replace the prevailing formulations and extend to the limit the available supply of soda ash.

It was necessary, of course, that the properties of any of the new glasses developed be such that they could be used in the established forming processes. The most important properties were the viscosity characteristics, liquidus temperature, and lack of opacity. It was also desirable not to alter radically the resistance properties of the glass.

Lithia was known to be a constituent that could minimize the need for soda, but the material is expensive and available only in limited quantities. Boric anhydride is also a good possibility, but it is considered an uneconomical constituent for glasses manufactured on a mass-production basis. Fluorides remained the only materials which were suspected of having good possibilities. These are available both as fluorspar and as slag constituents, and both are relatively inexpensive. The effects of small quantities of fluorine on the physical properties were studied and the results are reported in this paper.

Slags from phosphate operations contain not only fluorine, alumina, silica, lime, and alkali but also a significant quantity of phosphorus pentoxide. An investigation of this slag therefore was made and is reported here. A typical analysis of a phosphate slag from Sligo, Tennessee, was as follows: $SiO_2$ 38.5, $Al_2O_3$ 10.9, CaO 45.7, MgO 0.3, $Na_2O$ 0.3, $K_2O$ 0.9, $P_2O_5$ 1.1, $F_2$ 2.0, $Fe_2O_3$ 0.18, MnO 0.12, and S 0.16.

The use of fluorides and phosphates is a well-established practice in the production of opaque glasses. The following analysis is typical of a fluoride opal: $SiO_2$ 65.5, $R_2O_3$ 5.7, CaO 10.6, MgO 0.1, $Na_2O$ 12.9, $K_2O$ 1.3, and $F_2$ 5.9.

## II. Chemical Composition and Preparation of Glasses

The raw materials used in the preparation of the glasses were Ottawa sand, burned dolomite, Solvay soda ash, technical sodium phosphate, and sodium fluoride. The analyses of the materials which introduced small quantities of impurities into the glass were as follows:

| | Burned dolomite | Ottawa sand |
|---|---|---|
| $SiO_2$ | 0.6 | 99.7 |
| $R_2O_3$ | 0.3 | 0.2 |
| $SO_3$ | 0.6 | |
| CaO | 57.2 | |
| MgO | 40.1 | |
| Ignition loss | 1.2 | |

Approximately 4½ lb. of each glass were melted in a large covered platinum crucible in a gas-fired furnace. These glasses were stirred several times during the initial melting period and were poured, crushed, and remelted with additional stirring. The total time of melting and remelting was approximately 20 hours at 2600°F.

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† The work is the cooperative result of the following members of the research staff at the Owens-Illinois Glass Co.: F. R. Bacon, Paul Close, D. P. Hall, R. C. Kennedy, and V. C. Rees.

TABLE I

CHEMICAL ANALYSES AND DATA ON MEASURED PROPERTIES OF $F_2$-SUBSTITUTED GLASSES

| | Base-1 | FS-1 | FS-2 | FS-4 | Base 2 | FS-5 |
|---|---|---|---|---|---|---|
| $SiO_2$ | 73.3 | 72.2 | 71.7 | 70.0 | 73.5 | 72.4 |
| $R_2O_3$ | 0.1 | 0.2 | 0.1 | 0.2 | 0.1 | 0.2 |
| CaO | 6.0 | 6.0 | 6.0 | 6.1 | 7.0 | 7.0 |
| MgO | 4.2 | 4.2 | 4.2 | 4.2 | 5.0 | 5.1 |
| $Na_2O$ | 16.5 | 16.5 | 16.6 | 16.6 | 14.4 | 14.5 |
| $F_2$ | | 1.1 | 2.0 | 3.6 | | 1.1 |
| | 100.1 | 100.2 | 100.6 | 100.7 | 100.0 | 100.3 |
| | | | Liquidus temp. (°F.) | | | |
| | 1610 | 1605 | 1705 | 1875 | 1760 | 1800 |
| | | | Primary phase | | | |
| | Tridymite | Devitrite | Tridymite | Tridymite | Diopside | Diopside |
| Log viscosity (°F.) | | | | | | |
| 7.65 | 1280 | 1235 | 1195 | 1150 | 1320 | 1275 |
| 7.00 | 1345 | 1300 | 1260 | 1215 | 1385 | 1340 |
| 6.00 | 1460 | 1415 | 1375 | 1340 | 1500 | 1455 |
| 5.00 | 1615 | 1570 | 1525 | 1500 | 1655 | 1610 |
| 4.00 | 1830 | 1780 | 1740 | 1705 | 1860 | 1815 |
| 3.00 | 2145 | 2085 | 2030 | 1995 | 2165 | 2100 |
| 2.00 | 2635 | 2560 | 2475 | 2420 | 2635 | 2540 |
| Deformation temp. (°F.) | 1023 | 1010 | | | | |
| Coeff. of expansion $\times 10^{-7}$ | 97 | 95 | | | | |
| | | | Chemical durability | | | |
| % $Na_2O$ in $H_2O$ | 0.085 | 0.070 | 0.069 | 0.066 | 0.062 | 0.055 |
| % $Na_2O$ in $N/50$ $H_2SO_4$ | 0.077 | 0.070 | 0.073 | 0.091 | 0.035 | 0.027 |

The actual chemical analyses as well as all the measured properties of the fluoride glasses may be found in Table I. The analyses and all measured properties of the phosphate glasses are shown in Table II. The fluoride compositions were computed on the basis of an expected loss of one third of the fluorine present in the unmelted batch. No provision was made for the loss of $P_2O_5$.

The substitutions indicated in Tables I and II are $F_2$ for $SiO_2$, $P_2O_5$ for $SiO_2$, and $P_2O_5$ for $CaO \cdot MgO$.

Originally a series of glasses containing calcite lime and $P_2O_5$ were melted, but the volatilization of $P_2O_5$ from the surface and immiscibility in the temperature range where viscosity measurements are normally made precluded an investigation of the properties of this series of glasses.

## III. Properties Measured and Methods of Determination

The properties of the glasses that were determined were (1) density; (2) liquidus temperature; (3) coefficient of expansion; (4) deformation temperature; (5) viscosity; (*a*) fiber softening point and (*b*) high-temperature viscosity; (6) resistance to dilute acid; and (7) resistance to distilled water. The methods of determination were exactly the same as those used in work previously reported.[1]

## IV. Discussion of Results

The measured properties of the eleven glasses are shown in Tables I and II. The changes of the various log viscosity (equal to 3, 4, etc.) temperatures as a function of $F_2$ for silica substitutions in a $16^1/_2$% $Na_2O$ base glass are shown in Fig. 1. Figure 2 shows the same changes as functions of $P_2O_5$ for $SiO_2$ and $P_2O_5$ for $CaO \cdot MgO$ substitutions.

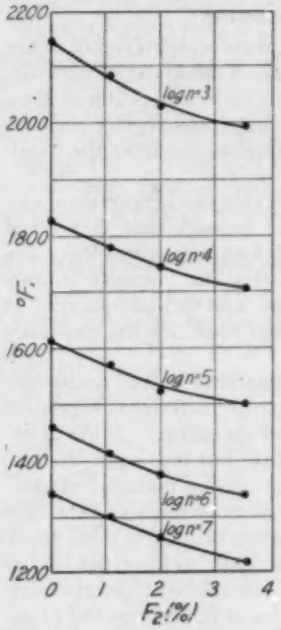


FIG. 1.—Effect of substitution of $F_2$ for $SiO_2$ on viscosity.

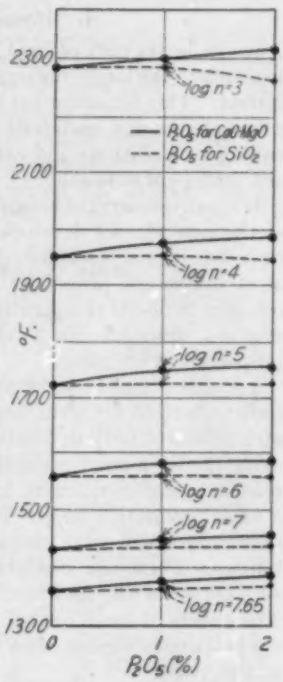


FIG. 2.—Effect of substitution of $P_2O_5$ for $CaO \cdot MgO$ and $SiO_2$ on viscosity.

[1] Owens-Illinois Glass Co. General Research Laboratory, "Effect of Barium Oxide and Zinc Oxide on Properties of Soda-Dolomite Lime-Silica Glasses," *Jour. Amer. Ceram. Soc.*, **25** [Feb. 1, No. 3] 61–69 (1942).

TABLE II

CHEMICAL ANALYSES AND DATA ON MEASURED PROPERTIES OF $P_2O_5$-SUBSTITUTED GLASSES

| | Base | PS-1 | PS-2 | Base | PD-1 | PD-2 |
|---|---|---|---|---|---|---|
| $SiO_2$ | 73.4 | 72.0 | 71.0 | 73.4 | 73.4 | 73.6 |
| $R_2O_3$ | 2.1 | 2.0 | 2.0 | 2.1 | 1.9 | 2.0 |
| CaO | 6.5 | 6.5 | 6.5 | 6.5 | 5.9 | 5.1 |
| MgO | 4.3 | 4.4 | 4.4 | 4.3 | 4.4 | 3.7 |
| $Na_2O$ | 12.5 | 12.7 | 12.7 | 12.5 | 12.4 | 12.4 |
| $K_2O$ | 1.1 | 1.1 | 1.1 | 1.1 | 1.0 | 1.2 |
| $P_2O_5$ | | 1.0 | 2.0 | | 1.0 | 2.0 |
| | 99.9 | 99.7 | 99.7 | 99.9 | 100.0 | 100.0 |
| | | | Liquidus temp. (°F.) | | | |
| | 1770 | 1790 | | 1770 | 1840 | |
| | | | Primary phase | | | |
| | Diopside | 1900 | 2090* | Diopside* | Tridymite | Opal 2020 |
| Log viscosity (°F.) | | | | | | |
| 7.65 | 1365 | 1365 | 1370 | 1365 | 1375 | 1385 |
| 7.00 | 1435 | 1435 | 1440 | 1435 | 1445 | 1455 |
| 6.00 | 1560 | 1560 | 1560 | 1560 | 1580 | 1585 |
| 5.00 | 1725 | 1725 | 1725 | 1725 | 1750 | 1755 |
| 4.00 | 1955 | 1955 | 1945 | 1955 | 1975 | 1985 |
| 3.00 | 2290 | 2290 | 2265 | 2290 | 2300 | 2315 |
| 2.00 | 2820 | 2820 | 2760 | 2820 | 2790 | 2835 |
| Deformation point (°F.) | 1075 | | 1025 | 1075 | | 1069 |
| Coeff. of expansion $\times 10^{-7}$ | 85 | | 88 | 85 | | 84 |
| Density (gm./cc.) | 2.4754 | | 2.4600 | 2.4754 | | 2.4257 |
| | | | Chemical durability | | | |
| % $Na_2O$ in $H_2O$ | 0.021 | 0.022 | 0.021 | 0.021 | 0.021 | 0.022 |
| % $Na_2O$ in $N/50$ $H_2SO_4$ | 0.020 | 0.019 | 0.020 | 0.020 | 0.022 | 0.025 |

* Immiscible.

### (1) Density

The replacement of 2% of $SiO_2$ by 2% of $P_2O_5$ causes a considerable decrease in density. Phosphorus pentoxide is one of the few materials that reduces the density of a glass when substituted for silica. The reduction is equivalent to 0.0077 gm. per cc. for each percentage of substitution. When CaO·MgO is replaced by $P_2O_5$, the change in density is of considerably greater magnitude, being equivalent to 0.0248 gm. per cc. for each percentage of substitution. Density measurements were not made on the fluorine glasses.

### (2) Liquidus Temperature

Table I gives the individual measurements of the liquidus temperatures of the glasses of the fluorine series. No generalizations can be drawn from the data. The first percentage substitution of fluorine for silica appears to shift the primary phase from tridymite to devitrite without changing the liquidus temperature. This may not be correct since the base glass composition lies very close to the tridymite-devitrite boundary, a region in which rapid changes in liquidus temperatures accompany a few tenths percentage variation in the silica content of a glass. Further replacement of $SiO_2$ by fluorine changes the primary phase back to tridymite and raises the liquidus temperature a considerable amount.

It should be noted that in spite of the fact that more than 3% of the silica of the base glass has been replaced by fluorine in a glass whose primary phase could have been altered by a replacement of a few tenths per cent of silica by either a dibasic oxide or by alkali, the primary phase is still tridymite. When diopside is the primary phase in a base glass, no phase change is observed when 1% of $F_2$ replaces 1% of $SiO_2$. The liquidus temperature raises 40° with this substitution.

No tendency toward opalescence or immiscibility is observed in the vicinity of the liquidus temperature in the glasses of the fluorine series.

When 1% of phosphorus pentoxide replaces silica in the base glass shown in Table II, the primary phase changes from diopside to tridymite without an appreciable increase in the liquidus temperature.

Glasses PS–1 and PS–2 show immiscibility in the temperature range where devitrification occurs. The temperatures at which the immiscibility ends are noted in Table II. The crystals of tridymite are found imbedded in the opaque glass of PS–1. No crystalline materials were found in sample PS–2.

The phase and liquidus temperature changes accompanying the replacement of CaO·MgO by $P_2O_5$ are shown in Table II. It is evident that the first percentage substitution changes the primary phase from diopside to tridymite and raises the liquidus temperature considerably. There is no evidence of opalescence in the first percentage substitution. The next percentage substitution causes the glass to become very opaque. The immiscibility ends abruptly at 2020°F.

### (3) Coefficient of Expansion

The coefficient of expansion of only one fluorine glass was determined, since it was evident after the first attempt that the specimens reacted with the fused-quartz plates of the interferometer. The difference between

the coefficients of this glass and its base is not of sufficient magnitude to be significant.

The substitution of $P_2O_5$ for $SiO_2$ raises the coefficient of expansion. When $P_2O_5$ replaces $CaO \cdot MgO$, there is little change.

### (4) Deformation Temperature

When 1% of fluorine replaces silica, the deformation temperature is decreased considerably. The temperature noted in Table I is that at which the reversal of fringe direction is observed in the interferometer. The curve from which this temperature is obtained shows a much broader range of temperatures over which no movement of fringes is noted other than is normal with soda-lime-silica glasses. It is suspected that this phenomenon is associated with the reaction of the quartz plates and specimens.

The replacement of either $SiO_2$ or $CaO \cdot MgO$ by $P_2O_5$ results in lower deformation temperatures; it is a considerable amount in the case of the silica substitution.

### (5) Viscosity

The effect of fluorine replacement of silica is shown graphically in Fig. 1, and the measured values are given in Table I. It is evident from Fig. 1 that the fluorine replacement has a profound effect on the viscosity of the glass. The data for the $16^1/_2$% base glass show that the substitution of 1% of $F_2$ for 1% of $SiO_2$ in the range of 0 to 2% of $F_2$ brings about an average lowering of 57°F. in the temperature for a log viscosity equal to 3. The average lowering of the log viscosity equal to 7 temperatures is 36°F. (0 to 3.6% $F_2$). In the 2 to 3.6% $F_2$ replacement range, the log viscosity equal to 3 temperatures is decreased at the rate of 22°F. for each percentage of substitution.

A comparison of the viscosity data of glasses base 2 and FS–5 in Table I indicates that the fluorine for silica replacement is just as effective in a lower alkali glass.

Table III compares the effect of 1.1% replacement of $SiO_2$ by $Na_2O$ and by $F_2$. It is evident that the fluorine is more effective in reducing the viscosity throughout the entire range than is soda. A lower log viscosity equal to 7 temperatures suggests that the fluoride glass will set up more slowly than the soda glass. This would necessitate somewhat reduced machine speeds. The change in deformation temperature previously noted indicates that a fluorine glass continues to be less viscous down to the 1000°F. region.

Figure 2 shows that the effect on the viscosity characteristics of a 1% substitution of $P_2O_5$ for $SiO_2$ is negligible. The next percentage substitution decreases the log viscosity equal to 3 and the log viscosity equal to 2 temperatures as shown in Table II. When $P_2O_5$ replaces $CaO \cdot MgO$, the viscosity curve is raised 10° to 15°F. throughout the range investigated.

### (6) Resistance to Dilute Acid

The effect of fluorine for silica replacement on the resistance to $N/50$ sulfuric acid can be followed in Table I. The first percentage substitution in the higher alkali base glass increases the resistance. Further substitution progressively decreases it. There is considerably increased resistance of the lower alkali glass for the first percentage of $F_2$ for silica substitution.

TABLE III

CHEMICAL ANALYSES AND VISCOSITY DATA ON $Na_2O$ AND $F_2$ SUBSTITUTIONS

| | Base | NS–1* | FS–1 |
|---|---|---|---|
| $SiO_2$ | 73.3 | 72.2 | 72.2 |
| $R_2O_3$ | 0.1 | 0.1 | 0.2 |
| CaO | 6.0 | 6.0 | 6.0 |
| MgO | 4.2 | 4.2 | 4.2 |
| $Na_2O$ | 16.5 | 17.6 | 16.5 |
| $F_2$ | | | 1.1 |
| | 99.9 | 100.1 | 100.2 |
| Log viscosity (°F.) | | | |
| 7.65 | 1280 | 1250 | 1235 |
| 7.00 | 1345 | 1320 | 1300 |
| 6.00 | 1460 | 1430 | 1415 |
| 5.00 | 1615 | 1580 | 1570 |
| 4.00 | 1830 | 1790 | 1780 |
| 3.00 | 2145 | 2100 | 2085 |
| 2.00 | 2635 | 2575 | 2560 |

* Extrapolated from laboratory data.

The acid resistance of the series of glasses wherein $P_2O_5$ replaces $SiO_2$ is not measurably changed. When $P_2O_5$ replaces $CaO \cdot MgO$, a decrease is noted.

### (7) Resistance to Distilled Water

A progressive increase in the resistance to distilled water accompanies the replacement of silica by $F_2$ in the $16^1/_2$% $Na_2O$ series; 80% of the increase occurs with the first percentage substitution. It is likewise evident that the resistance of the lower alkali base glass is improved by the replacement; $P_2O_5$ replacements for either $CaO \cdot MgO$ or $SiO_2$ are without measurable effect on this property.

## V. Conclusions

(1) Fluorine is very effective in reducing the viscosity of a glass and quite probably will increase the working range.

(2) Fluorine will increase the liquidus temperature rapidly in the base glasses investigated. The quantity used to replace soda must therefore be limited.

(3) Fluorine does not decrease the chemical resistance in the range used in these experiments.

(4) Fluorine compounds are probably vaporized above the annealing temperature of a glass.

(5) Phosphorus pentoxide has but little effect on the viscosity characteristics of the base glass used in this work when it replaces silica. The viscosity is increased when it is substituted for dolomite lime.

(6) Phosphorus pentoxide does not affect the resistance properties in the range investigated when it replaces either $CaO \cdot MgO$ or $SiO_2$.

(7) The tendency of glasses to become opalescent in the working range when phosphorus pentoxide is present suggests that the element is not desirable in soda-lime bottle glasses.

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# REFINED METHOD OF CONTROL OF CORDINESS AND WORKABILITY OF GLASS DURING PRODUCTION*

## Statistical Control Charts Applied to Daily Measurements of Density

By L. G. Ghering

Abstract

Many glass plants obtain daily measurements of the density or specific gravity of the glass in each furnace. Small daily fluctuations of about ±0.0010 density unit are usually taken for granted, while pronounced changes within a two- or three-day period are a matter of concern; but heretofore neither criteria of permissible variability nor rules for interpretation of the data have been in general use.

In the present work, the control-chart method of statistical analysis of past data has been applied to data from ten glass furnaces. Small daily fluctuations of density are found to be statistical in character, and the predominant cause of large variations is found to be in the batch house. The rational subgroup sample to be used in analyzing such variations and in operating a control chart is found to be a subgroup of three consecutive daily density values obtained from a particular furnace. Using this subgroup, the average 3-day range of density for the ten furnaces varied from 0.0006 to 0.0023, and the corresponding 3-sigma limits for daily variation from the central line density were ±0.0011 to ±0.0040. A typical value for the average 3-day range of density is 0.0012 and a value no larger than this is a reasonable goal for a glass container plant.

The use of control charts for maintaining a state of statistical control of density during production is illustrated for four furnaces over a 2- to 6-month period. Many assignable causes of variation were found in the batch house, usually in the scales; other assignable causes were changes in cullet and in raw materials, changes in firing of the furnace, and laboratory errors in measurement of the density.

Present experience indicates that it is difficult to maintain a state of statistical control with the types of batch-weighing equipment in use in some plants. The importance of control, however, was demonstrated for two furnaces in two different plants by the fact that cordiness increased with increasing 3-day range of density. When the density was not maintained under statistical control in one plant, trouble was experienced with checks in the ware.

The use of control charts for keeping lack of control within tolerable limits is discussed for one furnace where the variations were small and the control limits narrow. The range was held under control, but the density showed "trends" and went out of control. In this instance, the 3-sigma control limits for variation of daily values from the central line density were ±0.0011, corresponding to ∓0.09% replacement of lime by silica. Inasmuch as composition changes in excess of ∓0.09% are tolerable in the present state of the art, a modified control limit corresponding to a composition change of approximately ±0.25% is suggested, the corresponding density limits being ±0.0030. When the 3-sigma limits for density are less than this value, modified limits may be used, although the 3-sigma limits for range are retained. When the 3-sigma limits are greater than ±0.0030, it is most desirable to maintain strict statistical control, and efforts should be made to reduce the variability; otherwise there may be excessive cordiness and other difficulties in fabrication of the ware. In some instances, a reduction in variability will require major repair of batch handling and weighing equipment or a new batch-house weighing installation. Other subgroup methods and other sources of variability are also discussed.

Control charts on density are of practical utility to plants. "Assignable-cause" variations are easily distinguished from unimportant, normal variations. The use of 3-sigma action limits keeps investigation of fluctuations to a minimum, and sets trouble-shooting, when it is necessary, on the right track. The charts, furthermore, are a useful guide toward a permanent reduction of the variability. They should be helpful to management in striking an economic balance among tonnage pulled, glass quality, and capital expenditures for improvement of batch mixing and handling and other changes. The time required to maintain a chart for one furnace is about one day for past-data analysis, one minute each day for plotting, and not more than one day per month for current analysis, review, and adjustment.

* Presented at Glass Division Autumn Meeting, American Ceramic Society, Pittsburgh, Pa., September 14, 1944. Received September 28, 1944.

This paper constitutes a report of one of the researches sponsored by the Glass Container Association of America and was also presented at the meeting of the Pittsburgh

## I. Introduction

Many glass plants make daily measurements of the specific gravity or density* of a sample of glass from each furnace. The Archimedes method[1] for determining specific gravity is usually employed, although a few plants have recently used a sink-float method[2] utilizing heavy liquids and standard density samples. Such measurements are widely used for control purposes, in lieu of chemical analyses, for several reasons, (1) necessary equipment can be easily obtained by a small plant laboratory, (2) specific gravity or density variation is a more sensitive and precise indication of composition changes than chemical analyses,[1] and (3) the measurements require little time, thus making it practicable to obtain a value each day on each furnace. The most recent practice is to make chemical analyses only to determine the particular combination of oxide changes that produce a known density change—in other words, as a trouble-shooting tool to find the necessary batch change or the difference between the calculated (theoretical) and the observed composition.

In the writer's opinion, however, the fullest use has not been made of the density data. One of the reasons lies in the complex relationship between density and composition. Many laboratories regard density changes with undue suspicion because of the uncertainty of the corresponding composition changes. Others have failed to find any correlation between density, or density variation, and important functional qualities of the glass, or ware, or components of the fabrication process.

When a density change occurs, it indicates a change in composition provided the annealing remains constant† although there may be rare instances where the converse, that is, no density change, no composition change, is not strictly true. Theoretically, there may be composition changes which leave the density unchanged; for example, in most container glasses the addition of 0.50% by weight of $Al_2O_3$ plus 0.47% $SiO_2$ produces a density change of 0.0000. It is the writer's experience, however, and this has been supported by others, that in glasshouse practice the danger of complete composition-density compensation is so remote that it may be virtually disregarded in practical plant operation.

It is also true that there are many other variables, aside from composition changes, that enter into density data and tend to detract from its value for making broad interpretations and analyses. Annealing has already been mentioned. Densities of ten lumps from various parts of the same bottle, in extreme cases, may vary by as much as ±0.0005 due to annealing differences or composition heterogeneity (cord). Furthermore, the measuring technique is subject to various errors, (*a*) inclusion of bubbles, seeds, or cracks in the sample, (*b*) dirty samples, (*c*) bubbles of air sticking to the immersed sample, (*d*) arithmetical errors, and (*e*) other experimental error. Then too, if absolute values, as distinguished from relative values, are being calculated, great care must be taken in temperature corrections, air buoyancy corrections, and calibration of weights.

But the foregoing limitations of the density method are often exaggerated. The important thing for control purposes is precision (reproducibility) as distinguished from accuracy. When measurements are reasonably accurate and reasonable care is taken with respect to annealing, density data do give an over-all indication of composition changes caused by batch scales, batch-scale settings, mixing losses, batch segregation, melting segregation, convection currents in furnace, furnace temperature and firing conditions, stagnant glass in active stream, volatilization and dusting loss, refractory contamination, changes in tonnage pulled, composition of foreign cullet, and others. When an unusual density variation occurs, a chemical analysis can be made if necessary in order to find the immediate cause of the variation. In the past, there has been fair success in using density data in lieu of chemical analysis for plant control, and this has been accomplished without the use of general rules for interpretation of the data. Although there are no published yardsticks for judging furnace or batch-house performance on the basis of day-to-day density variations, a 3-day density spread of 0.0055 has been illustrated‡ as "poor performance" and 0.0015 as "good performance."

Good density data may be had by choosing, each day at the same hour, a bottle of approximately the same grade of annealing from the same shop and lehr position (preferably a heavy-pull shop) and by selecting a seed-free sample from the shoulder of the bottle. The fact that many plants accumulate density data implies that their value justifies the effort. The writer's main thesis is that if the data are worth taking they are worth analyzing by the relatively simple control-chart procedure outlined herein.

In the following pages, the powerful tool of statistical control charts[3] has been applied to density data. The

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Quality Control Society, Pittsburgh, September 23, 1944.

* In reporting data it is essential, of course, to distinguish between density and specific gravity. The term density (in gm. per cc. at 20°C.) will be used in this paper; density in gm. per cc. at 20°C. equals specific gravity at 20°C. × 0.99820, the density of pure water at 20°C.

[1] "Glass Composition Control by Use of Specific-Gravity Determinations," *Glass Ind.*, **22** [9] 384–86 (1941); *Ceram. Abs.*, **21** [1] 8 (1942).

[2] L. G. Ghering and M. A. Knight, "Properties and Diagnosis of Cords in Pressed and Blown Glassware," *Jour. Amer. Ceram. Soc.*, **27** [9] 260–66 (1944); the sink-float method recently used is the one mentioned briefly under subtitle VI (3), p. 263; the heavy liquids and their temperature-density factor are given under subheading (4) p. 264. The adaptation of this and other techniques to lump-density measurements in plant laboratories is under study in this laboratory at the present time.

† For the effect of annealing on density, see reference (2) Table I, p. 261.

‡ See Fig. 2 of reference (1), p. 385.

[3] (*a*) "Control Chart Method of Controlling Quality During Production," American Standards Association, ASA Designation Z1.3–1942 (29 West 39th Street, N. Y.; the small booklet is easily obtainable at a nominal cost).

(*b*) Leslie E. Simon, An Engineer's Manual of Statistical Methods. John Wiley and Sons, Inc., New York, 1941.

(*c*) W. A. Shewhart, Economic Control of Quality of Manufactured Product. D. Van Nostrand Company, Inc., New York, 1931.

TABLE I

GLOSSARY OF SYMBOLS AND TERMS*

$n$, number of consecutive daily density values in a group.
$R$, range (maximum spread) of values in subgroup sample of consecutive daily density values; e.g., for the 3 density values 2.5004, 2.5019, and 2.5016, the range is 0.0015.
$\bar{R}$, average range of a number of subgroup sample ranges.
$\sigma$, sigma; standard deviation.
$X$ individual value (daily density measurement).
$\bar{X}$, average of a subgroup sample.
$\bar{\bar{X}}$, average density over a period of 90 or more days; central line density.
$UCL_X$, 3-sigma upper control limit for individuals.
$UCL_{\bar{X}}$, 3-sigma upper control limit for subgroup averages.
$LCL$, lower control limit (3-sigma).
Central line, either average density, $\bar{\bar{X}}$, or average range, $\bar{R}$, over a period.
Dispersion, variability or spread of the individual values of a universe.
Extension, end of past data analysis, projection of center lines and limits, and beginning of operation of the control chart.
Level, average density for a particular period (see ‡ footnote, section VI (1)).
Theoretical (composition), average of those chemical analyses of samples in which the glass density is close to central-line density; or batch-calculated composition adjusted for furnace losses, etc.
Weighted mean (composition), same as theoretical.
Weighted mean (density), representative central line density for those periods when chemical analyses are close to theoretical.
Universe, large group of values (densities), essentially alike.

* The control chart symbols and terms are the same as those used in the ASA pamphlet, reference (3*a*).

3-day subgroup range of density described gives a definite measure of performance, and 3-sigma control limits indicate when an investigation of causes of variations is justified. Sampling is related to expected variations in advance so that when lack of control is indicated, one knows where to look for the trouble; investigation or trouble-shooting, when necessary, is kept on the right track. This method of control serves as a preventive of certain types of serious cordiness as well as a method of preventing composition changes of sufficient magnitude to change the workability of glass in the forming machines. It will be shown that, in general, this method of plant control is more practical as well as more precise than chemical analyses.

## II. Data

All original data* on composition, density, and ware grading are given in chart form in Figs. 1 to 12. They are believed to be fairly typical of over-all performance in the glass-container industry and include data from five plants. The figures and their legends give most of the detail and, if they are studied in numerical order, give a fairly complete story independently of the text of the paper.

A glossary of the symbols and terms used in the figures and in the text is given in Table I.

The partial correlation of ware grade (for cord) with 3-day range of density given in Fig. 1 is not an essential part of the control-chart method, but the fact that some correlation exists enhances the value of the control chart and is concrete evidence that control of density is an important objective.

A case history of poor measurements (Archimedes method) and subsequent improvement in laboratory technique is given in Fig. 2. This illustrates the utility of control-chart analysis when errors in the measurements themselves are suspected.

## III. Control-Chart Method of Analyzing Variations

### (1) Fundamental Considerations in Selecting a Rational Subgroup

In any control-chart analysis, useful information is obtained when the data are subgrouped consecutively in the order of production. It is essential that the subgroup samples be related to the component parts of the process. Then, if unusual variations are found, it is relatively easy to identify the component that caused the variation. As will be seen later, the main components of furnace-glass density data, that is, the main factors affecting the density values, are the raw materials, the batch house, the furnace, and the laboratory measurement itself.

### (2) Choice of 3-Day Subgroup Sample

Inspection of the typical daily density curve in Fig. 4 (solid line, daily density) and also of the daily density curves in Figs. 6 and 7 indicates that daily densities are essentially statistical in character. Furthermore, inspection of the daily density curves in Fig. 3 for three furnaces on the same batch, raw materials, and batch house indicates that the predominant day-to-day fluctuations are characteristic of the batch house and not of the particular furnace. A statistical measure of this batch-house fluctuation is needed. Mean deviation from the mean (absolute values), standard deviation (of subgroups or of individuals), and the average range of subgroup samples are available statistical measures of dispersion. The use of the average, $\bar{R}$, of the subgroup sample ranges, from which the standard deviation, sigma, may be calculated by using a simple factor, is the standard control-chart procedure.[3(a)] The remaining problem is whether the choice of subgroup size shall be 2, 3, 4, 5, 6, or more consecutive daily density values from the particular furnace. The use of a group as small as 2 is not good statistical procedure and one larger than 4 gives undue weight to trends.†

* Because of the confidential nature of plant operating data, each set of ordinates for composition and for density has been changed by some fixed amount on most charts. Inasmuch as the spread or range of the data has not been changed, their essential statistical character remains intact.

The author gratefully acknowledges the aid of those companies and individuals who provided the data, assisted in the detailed analysis thereof, and helped to make the control chart application a success.

† Strictly speaking, data which show obvious trends are not well suited to computations of sigma in that the calculated value of sigma is somewhat too large and the 3-sigma limits are only approximately correct. This does not seriously affect the practical application to the kind of data illustrated. In this connection, see section VII, (3) and (5), pp. 386–87.

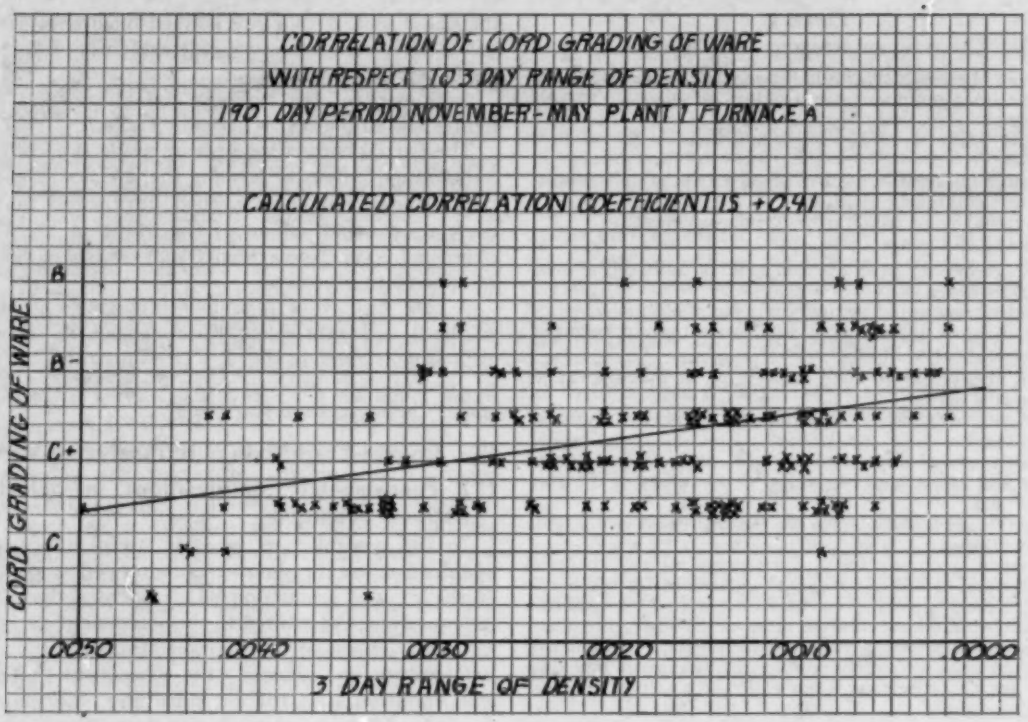



FIG. 1.—Ware grade plotted as a function of 3-day range of density, i.e., the maximum spread of three consecutive daily density values; from line of best fit, calculated by least squares method, cord grading seems to improve with decreasing range of density; 190-day period November to May, plant No. 1, furnace A; calculated correlation coefficient is +0.41.

In preliminary investigations, ranges were determined for subgroup samples of both 3 and 4 daily density values. When subgroup samples of 4 were taken, the range on a particular day for the subgroup sample including that day and the three previous days did not correlate well with the ware grade for cord on this particular day. Although the correlation coefficient was not calculated, inspections revealed that it was near zero; also, the observation is often made in the plants that the density level on the fourth day back is too remote to be reflected as a cause of "today's" cordiness. On the other hand when subgroups of 3 daily density values were used, a correlation between a 3-day range of density and ware grade for cord was found, the cord quality improving with decreasing values of the 3-day range. The calculated coefficient of correlation (method of least squares) was 0.41 for furnace A and about 0.35 for furnace B in plant 1. In the third case where data were available, namely, furnace A in plant 3, the coefficient was 0.27. Because of this partial correlation, a subgroup sample of three consecutive daily density values was adopted. In Figs. 4 to 9, the range and ware-grade charts are located one above the other to aid the eye in following this functional relationship.

The correlation of 0.41 in Fig. 1 is fair partial correlation. Actually, a correlation approaching 1.0 cannot be expected since the ware grade is not a grading for cord only; it gives weight to the particular location of the cord in the ware, for example, tension cords on the outside surface are heavily weighted. Furthermore, it is a subjective grading rather than a measurement. Even more important is the fact that day-to-day batch-house variation, the main factor in 3-day range of density, is only one of the several causes of cordiness; other causes are refractory corrosion, stir-up of stagnant glass on the bottom of the furnace, and batch segregation.

### (3) Calculation of Range

The method of computing range from a subgroup sample of three consecutive daily-density measurements is illustrated in column (5) of Table II, the range of a subgroup being the maximum spread of the subgroup values. In analyzing past data, it is best to compute the ranges for 30 to 60 subgroup samples representing three to six month's production and then determine the average, $\bar{R}$. This is the measure of variability and the key to the control-chart method and from it the control limits are computed.*

For the 3-day range curves in the figures, a range is obtained each day by using the density on a particular day along with the densities on the two previous days as a 3-day subgroup sample.

* After finding $\bar{R}$, compute its upper 3-sigma control limit by the formula $UCL = 2.574\ \bar{R}$. If any $R$'s going into the $\bar{R}$ exceed the $UCL$, designate them as mavericks, throw them out, and recompute $\bar{R}$, especially if the cause of the maverick can be accounted for, as something apart from the normal process; then the recomputed $\bar{R}$ becomes the representative yardstick of the normal process (see reference 3(*b*), pp. 55 and 56).

TABLE II

DETERMINATION OF $\bar{R}$ AND 3-SIGMA LIMITS AND ILLUSTRATION OF SUBGROUP METHODS FOR DAILY DENSITIES
PLANT NO. 4 FURNACES A, B, AND C

| (1) | (2) | (3) | (4) | (5)* | (6)† | (7)‡ |
|---|---|---|---|---|---|---|
| | Daily Density | | | | Subgroup range | |
| May | A | B | C | $n$ = 3 days A | $n$ = 6; 2 days A, B, C, | $n$ = 3; 1 day A, B, C, |
| 16 | 2.4918 | 2.4923 | 2.4920 | | | 5 |
| 17 | 13 | 10 | 09 | | 14 | 4 |
| 18 | 10 | 11 | 09 | 8 | | 2 |
| 19 | 05 | 08 | 09 | | 6 | 4 |
| 22 | 00 | 04 | 03 | | | 4 |
| 23 | 00 | 09 | 10 | | 10 | 10 |
| 24 | 09 | 09 | 02 | 9 | | 7 |
| 25 | 40 | 33 | 30 | | 38 | 10 |
| 26 | 25 | 29 | 30 | | | 5 |
| 27 | 35 | 23 | 31 | 15 | 12 | 12 |
| 31 | 30 | 28 | 26 | | | 4 |
| June | | | | | | |
| 1 | 42 | 39 | 38 | | 16 | 4 |
| 2 | 40 | 47 | 43 | 12 | | 7 |
| 3 | 36 | 25 | 43 | | 22 | 18 |
| 5 | 30 | 32 | 36 | | | 6 |
| 6 | 49 | 48 | 45 | | 19 | 4 |
| 7 | 28 | 30 | 32 | 21 | | 4 |
| 8 | 30 | 35 | 35 | | 7 | 5 |
| | Avg. range $\bar{R}$ for data | | | 13 | 16 | 6.3 |
| | Avg. range April 5–June 8 | | | 12 | 12 | 5.8 |
| | Range factor | | | 2.574 | 2.004 | 2.574 |
| | Range upper control limit | | | 31 | 24 | 15 |
| | ±3-sigma limits, individuals ($X$) | | | ±22 | ±14 | ±10 |
| | ±3-sigma limits, subgroups ($\bar{X}$) | | | ±12 | ± 6 | ± 6 |

* Homogeneous subgroup; separate chart for each furnace indicates day-to-day batch-house variability; overlapping ranges are not shown.
† Nonhomogeneous (stratified) subgroup; improper method; limits too narrow.
‡ Inapplicable if day-to-day batch-house variation is more important than furnace differences.

### (4) Setting Up Control Limits and Central Lines

Having found $\bar{R}$, the 3-sigma control limits for both the range chart and the density chart are computed from factors.* The control limits for density shown in the figures are for individual values, that is, daily densities, except those in Fig. 5, where both subgroup and individual limits are shown. The central line, $\bar{X}$, on the density chart is the average density for the past 90 days or so, mavericks, that is, assignable causes accounted for, having been eliminated. Judgment is necessary in selecting a period for the central line which is representative of a given grade of annealing, raw materials, batch formula, scale settings, etc.

### (5) Significance of 3-Sigma Control Limits

The $\bar{R}$ and the 3-sigma limits have been derived from past performance of the process. The batch house has, in effect, revealed what it is normally capable of doing. It cannot be expected to do any better without making changes (different raw materials, better batch system, etc.), but it should stay within its control limits. The probability of a limit being violated is only one or two chances in 1000, unless something unusual has occurred. When the density or range value falls outside a limit, that is, goes out of control, there is an assignable cause which can be found and eliminated. This is the crux of the control-chart method.

The 3-sigma limits, of course, include all variables in the process, for example, annealing, measurement errors, and all functional components of the batch, batch house, and furnace that affect density. Most of these variables were mentioned in the introduction (section I). Many of them operate, and they are not explicitly separated. It is for precisely this reason that a statistical treatment is advantageous. A yardstick is provided which indicates when it is worth while to take action and when it is not.[4]

## IV. Analysis of Past Data

### (1) Poor Precision of Laboratory Measurements

In Fig. 2 is shown a case of variability of density values due to the poor precision of the laboratory measurements. The analysis was made in August, several months after the data were taken. The supervisor's records, however, showed that about February 1 it was suspected that there were errors due to lack of skill on the part of the technician, and the latter was given instruction as to the proper technique. The

* For formulas and factors for various subgroup-sample sizes, see reference 3(*a*), pp. 37, 38, and 39. For a sample size of 3, the following formulas apply:

| Chart | Central line | 3-sigma control limits |
|---|---|---|
| Ranges | $\bar{R}$ | 2.574 $\bar{R}$ and 0 |
| Subgroup avg. | $\bar{X}$ | $\bar{X} \pm 1.023\bar{R}$ |
| Daily values | $\bar{X}$ | $\bar{X} \pm 3\bar{R}/1.693$ |

[4] J. M. Juran, "A, B, C of Quality Control," *Glass. Ind.*, 25 [8] 358; [9] 407 (1944).

(1944)

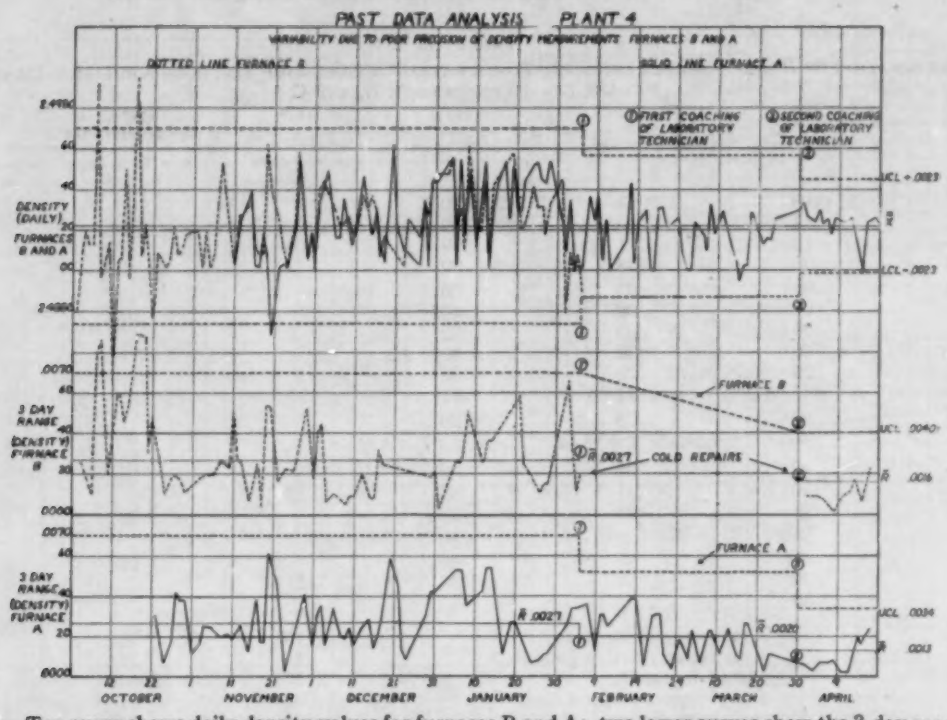



FIG. 2.—Top curve shows daily density values for furnaces B and A; two lower curves show the 3-day ranges plotted separately: A significant change due to improvement in laboratory technique occurred February 5, and the ranges were recalculated. These new ranges and the corresponding limits extend to March 30, when another similar change occurred. The third set of ranges and limits computed extends to August 31, as shown in Fig. 3.

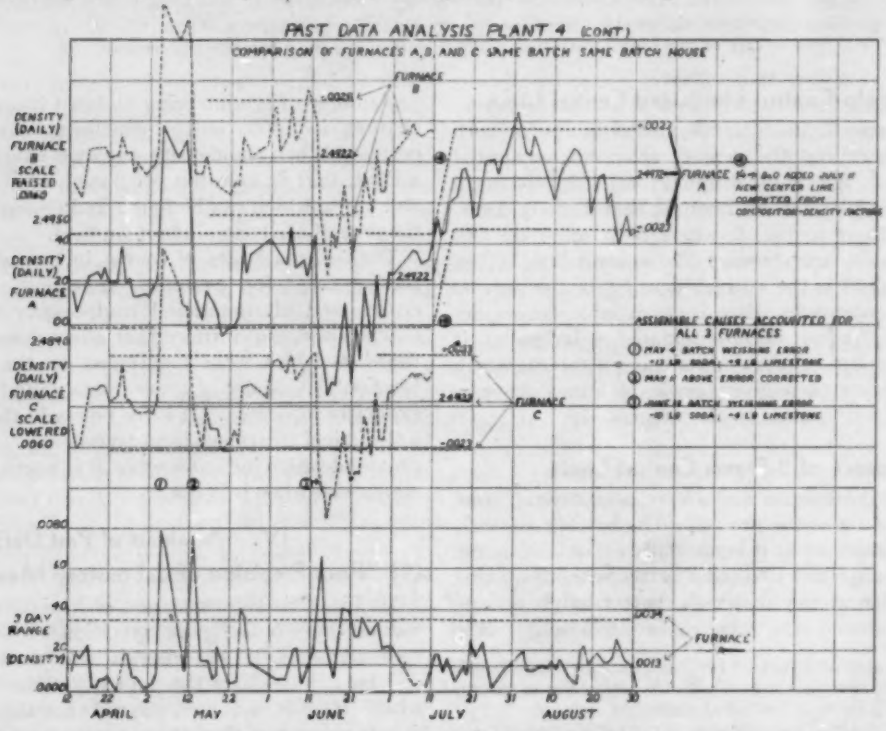
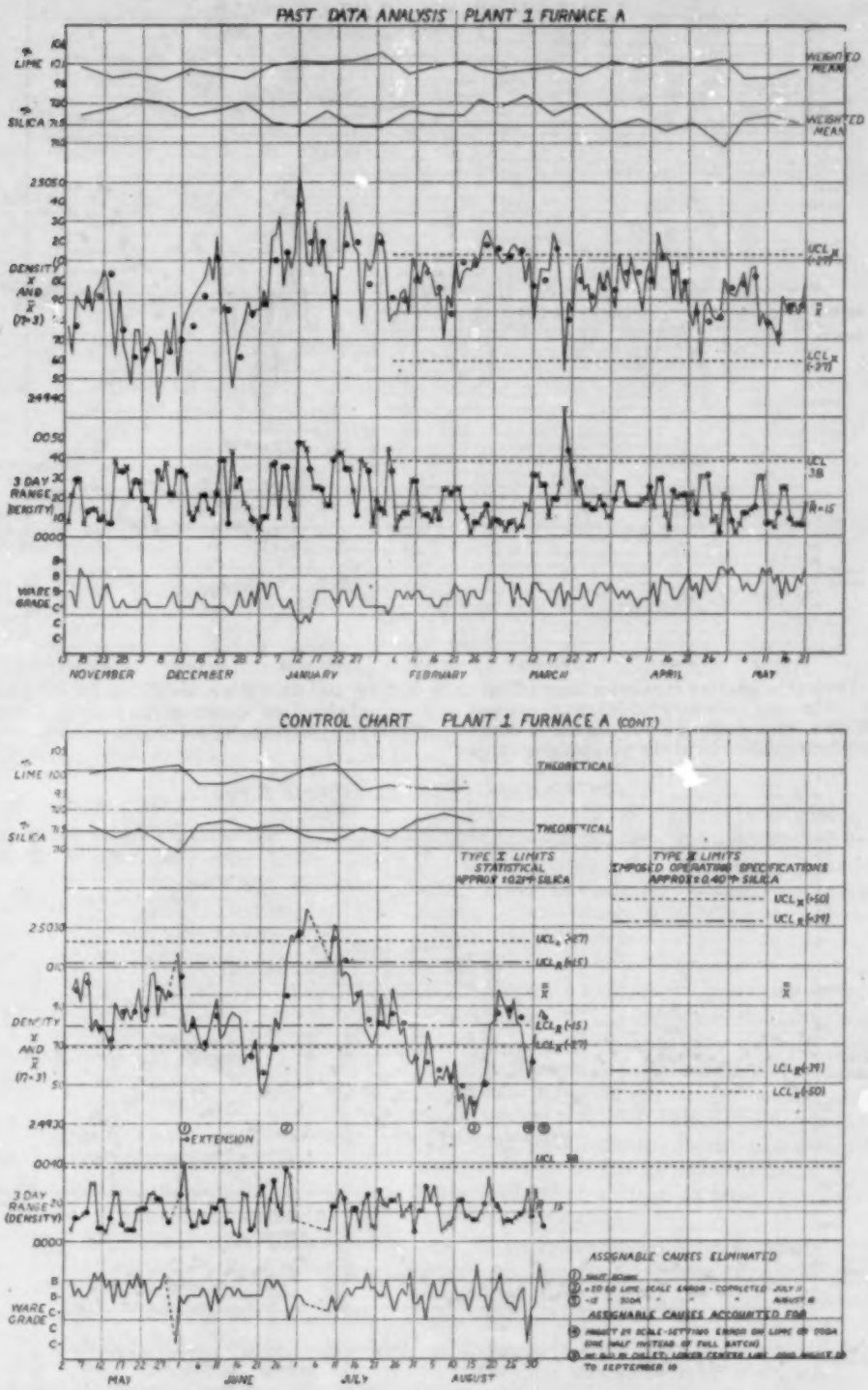

PAST DATA ANALYSIS PLANT 4 (CONT.)
COMPARISON OF FURNACES A, B, AND C SAME BATCH SAME BATCH HOUSE
DENSITY (DAILY) FURNACE B SCALE RAISED .0060
DENSITY (DAILY) FURNACE A
DENSITY (DAILY) FURNACE C SCALE LOWERED .0060
3 DAY RANGE (DENSITY)
ASSIGNABLE CAUSES ACCOUNTED FOR ALL 3 FURNACES
APRIL
MAY
JUNE
JULY
AUGUST

FIG. 3.—Furnaces B and A, as well as furnace C, from date of April 12. In order to separate the three daily density curves, the curve of *B* is raised 0.0060 scale unit and that of *C* is lowered an equal amount. A batch change for furnace A on July 11 is shown with the corresponding change in the central line. Three assignable causes are indicated. These are accounted for, in retrospect, from detailed records that accompanied the data. The bottom curve is the range curve of furnace A only.




FIGS. 4 and 5.—Curves for lime and for silica, for daily density ($X$), average subgroup density ($\bar{X}$), 3-day range of density, and for ware grade. The crosses on the range curve denote overlapping ranges. The period from November 13 to May 21 is shown in Fig. 4, and that from May 2 to September 2 in Fig. 5 (nineteen days overlapped). The average range and the average density with its corresponding 3-sigma limits for daily values are shown as well as limits for 3-day subgroup averages, $\bar{X}$, and alternative type II limits for both daily density and subgroup averages. (For the limit values given, the 0.00 has been omitted.) Assignable causes are indicated and classified into two groups, those eliminated and those merely accounted for; the limits and central lines are extended from June 1, the beginning date of actual operation of the chart as a control chart.

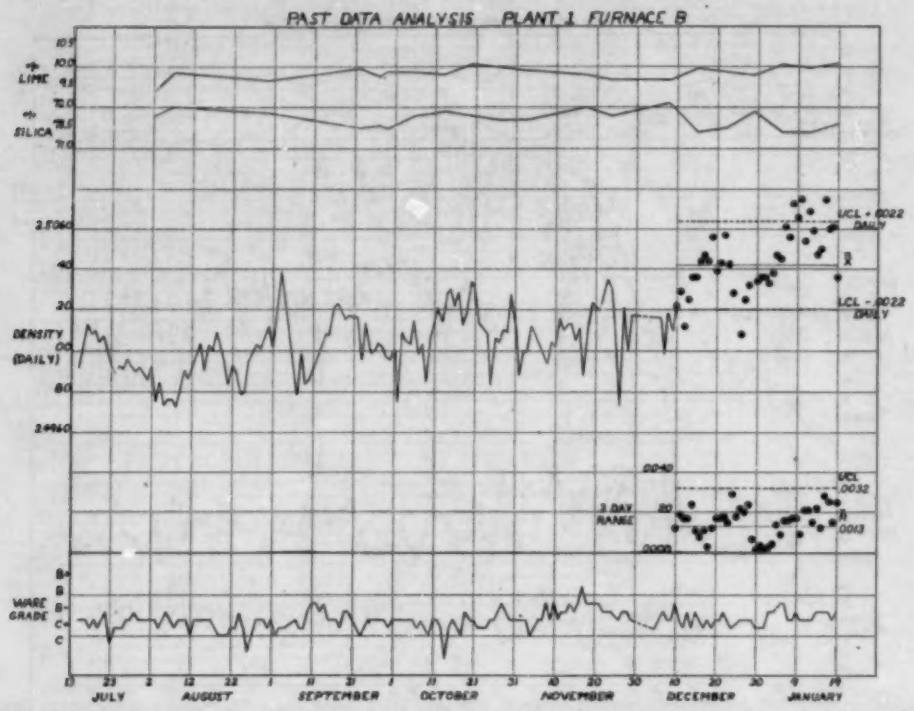



FIG. 6.—Typical laboratory curves for lime, silica, daily density, and ware grade are shown for the period July 13 to December 10. The only necessary additions to convert to a control chart are shown on the right. The change from a broken line to dots is not essential, but the use of dots is customary in plotting control charts. Conventional subgroup ranges are not distinguished from the overlapping ranges.

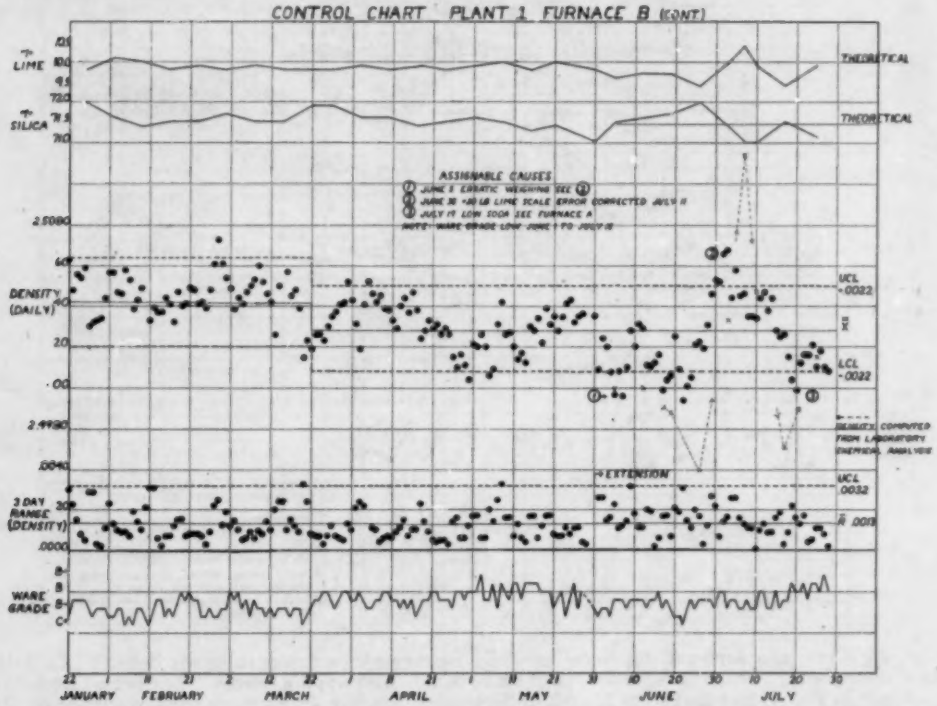



FIG. 7.—$\bar{R}$ was computed for the period December 10 to May 31 and the first central line calculated for the period December 10 to March 22. A new central line was calculated for the period March 22 to June 1. On June 1, the 3-sigma daily limits were extended, and the chart was put into operation. Assignable causes and their corrections are shown. The crosses shown in the period June 5 to July 25, some of which are connected by thin dotted lines, are densities calculated from the chemical analyses for comparison with the measured densities.

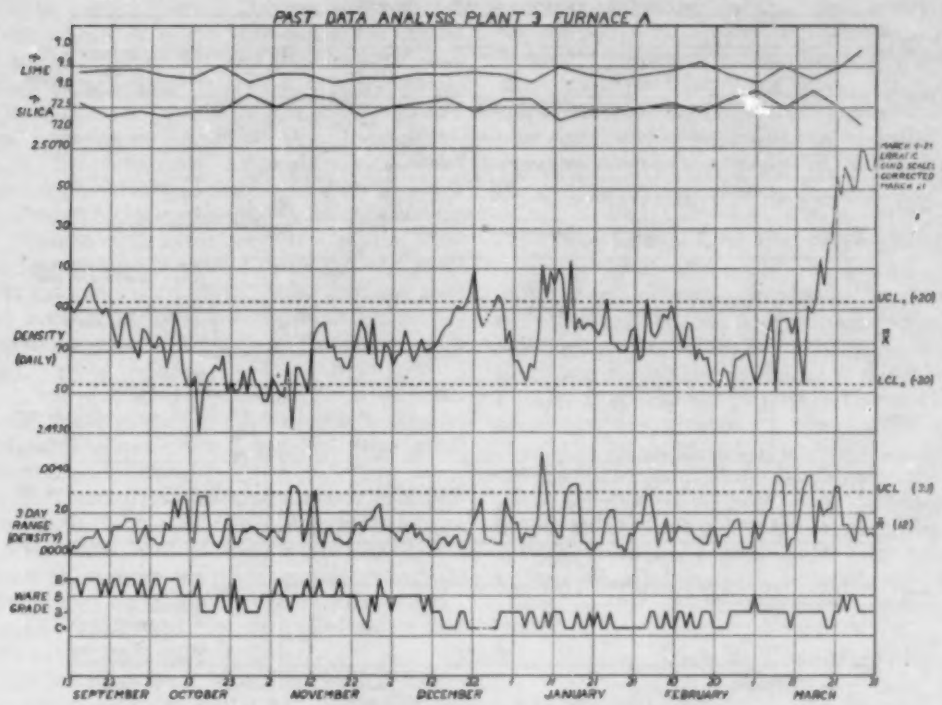



FIG. 8.—Curves for lime, silica, daily density, 3-day range of density, and for ware grade from September 13 to March 31. Analysis of the data up to March 11 gave an average range, $\bar{R}$, of 0.0012. The corresponding central lines and limits are drawn in. An assignable cause, accounted for on March 4, is indicated.

average range for furnace A decreased from 0.0027 to 0.0020. This variation was still rather high, and so on April 1 the technician was coached a second time. The average range for the smaller furnace B then decreased to 0.0016, and for furnace A to 0.0013, computed for the period April to August (see also range curve in Fig. 3). This case history illustrates a situation where the density variation was apparently high, but nonetheless for the most part was under control. The control chart is useful in that it gives a definite measure of the variation during the three periods. The changes in $\bar{R}$ and in the limits are consistent with the causes noted in the past records.

## (2) Assignable Causes Accounted For

One important use of control charts is that of analyzing causes of variation. In the previous example, the cause was known and the control chart contributed little new information. Other important applications, however, are illustrated in several figures bearing the title "Past-data analysis." The causes of variation were not so easily accounted for in these cases.

In Fig. 3 are shown three assignable causes corresponding to batch weighing errors noted in the past records. There is also a change in level on furnace A owing to the addition of BaO to the batch.

In Fig. 4 is illustrated a more elaborate method of plotting the data. The 3-day range is plotted as a dot on the third day of the subgroup sample while the crosses represent overlapping 3-day ranges. Thus the range chart is complete, with a point recorded each day. The average of each 3-day density subgroup, $\bar{X}$, is also plotted as a dot (on the third day) but the overlapping $\bar{X}$'s are not included, since they serve no particular purpose. A lack of control is indicated for both range and density. Since the rational subgroup sample of three consecutive daily density values was taken in order to reveal day-to-day batch-house variations, the assignable causes probably were in the batch house, but the incomplete past records did not account for them.

In Fig. 8 is shown essentially the same information, the difference being that $\bar{X}$'s are not shown on the density chart. Overlapping ranges on the range chart, moreover, are not distinguished from the conventionally segregated subgroup ranges. One assignable cause is accounted for by past records. In Fig. 9 beyond April 2, the variability appears to have decreased and the range was recalculated.

On the right-hand portion of the chart in Fig. 6, the dots representing the daily density values are not connected by lines. The same scheme is followed on the range chart. This is the most usual form of control chart but, of course, it is not necessary for glass houses to change their present practice of drawing in connecting lines. The main portion of Fig. 6 illustrates a typical glasshouse density chart, while the extreme right illustrates the only necessary additions to make it a control chart, and, as just stated, it is not even necessary to switch to dots. In the continuation of the chart, in Fig. 7, a change in level is indicated about March 22. This represents an assignable cause in raw materials or batch weighing.

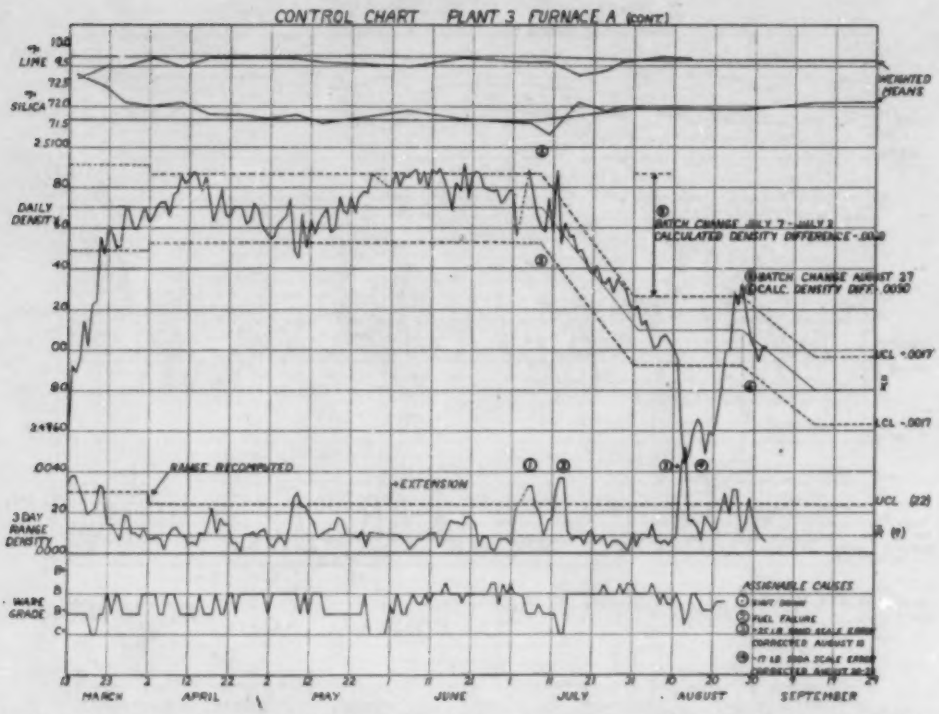



FIG. 9.—Curve begins on March 12 (18 days overlapping) with a new central line for density, drawn in at the higher level of the period April 2 to June 1. Since the range appeared to have decreased after April 2, a new range and corresponding limits were calculated for this period. The chart was put into operation on June 1. Several assignable causes indicating a lack of control of both range and density are noted, and two batch changes with accompanying changes in the central line of the density curve are indicated. The new central lines for density level were calculated in advance by the use of density difference factors.

### (3) Role of Furnace Size

Where furnaces of different sizes are operating on the same batch and batch house, $\bar{R}$ depends somewhat on the size of the furnace. For example, in plant 1, the relatively large furnace B has an $\bar{R}$ of 0.0013 whereas that of the smaller furnace A is 0.0015. Data for all furnaces are given in Table III, p. 387. In plant 4, the corresponding figures are 0.0013 and 0.0016. These findings on furnace size substantiate the common observation that the smaller furnace in a plant is more sensitive to changes and the glass is more apt to be cordy. Thus it is seen that the 3-day subgroup of a particular furnace is not entirely a measure of day-to-day batch-house variation. The explanation probably lies in the fact that the large back-log of molten glass in the larger furnace cushions or dampens the day-to-day batch-house variation. There appears to be no way to separate the variable of furnace size and that of day-to-day batch-house variation except to plot two charts, one with a subgroup sample (of densities) of three consecutive days on a particular furnace and the other a sub-group sample including all furnaces on a particular day (see Table II, column (7)).

### (4) Related Quality Characteristics; Glass Workability

In Table III, columns (7) and (9) give further data supporting the wisdom of using the 3-day range as a measure of variation and as an analytical tool. The cord-quality characteristic of a particular batch house correlates well with the average 3-day range and the condition of the batch-weighing equipment.

In one plant, moreover, it was noted that "checking" (tiny cracks on the surface of the ware) occurred when the density level was out of control (see discussion of Fig. 5, section VI (2)).

In glass plants, there are frequent complaints from the hot end that the glass is not working properly on a particular day. The laboratory usually is unable to find the cause; chemical analyses do not give a definite clue. Evidently the forming machine is more sensitive to changes in composition than analytical procedures; someone has said that the modern forming machine is glass technology's best viscosimeter. Therefore, the question is now raised as to whether or not the 3-day density range correlates with workability of glass. The difficulty in answering this question lies in measuring those factors which affect the machine. This suggests trying the correlation of number of checks in off-ware, poor seams, distribution, or total off-ware, etc., with 3-day density range. It should be worth trying. The 3-day range of density may turn out to be an analytical tool more powerful than is indicated by the present discussion.

## V. Maintaining Control During Production

### (1) Locating and Eliminating Assignable Causes

The maintaining of a state of statistical control is

another use of control charts. Having analyzed past data and set up control charts with limits, the central lines and limits are extended and the chart put into operation. The control charts in Figs. 5, 7, and 9 were put into operation on June 1, at the points marked "extension"; the one in Fig. 10, at the beginning date.

In Fig. 5, five assignable causes were identified.* Three were accounted for and eliminated while two were merely accounted for, one of the latter being a change in cullet. Of those eliminated, one was a holiday shutdown and two were scale errors. Although statistical control was not maintained during June, July, and part of August because of various circumstances, it was maintained in August and September (the latter month not shown).

In Fig. 7, where another furnace on the same batch house as the above is shown, the situation is similar. Three assignable causes were found and eliminated; all of these were scale errors.

In Fig. 9, four assignable causes and two batch changes are indicated. One of the assignable causes was a holiday shutdown, another was a major fuel failure where subsequent high temperature stirred up heavy bottom glass, and the other two were scale errors. A fair degree of statistical control was achieved and continued through September (not shown).

In Fig. 10 is an example of narrow control limits due to the low 3-day range of 0.0006. Although the range was under control, there are trends in the density values and six assignable causes, one of which was a change in raw materials, namely, sand. All of the assignable causes represented minor composition changes of not more than $\mp 0.20\%$ silica replacing lime. This situation is discussed fully in section VI.

It is interesting to note that most assignable causes were batch-house weighing errors. These assignable cause variations were not due to fluctuations of the annealing of the sample or some of the other variables that are, somehow, always subject to laboratory suspicion. The present work seems to indicate that reannealing of the sample is not necessary, if reasonable precautions are taken. The other known variables which did not show up as assignable causes are obscured behind the statistical maze of normal variations; they need not be considered, at least not until after the more flagrant offenders have been found and eliminated.

The facts that there were so many assignable causes, that delays occurred in identification and correction, that the density was out of control some of the time, and that there is evidence of trends indicating poor control even when the density was within the limits do not necessarily detract from the value and usefulness of limits. Rather, the fact that the assignable causes were easily identified in most instances and that the density came back into control when corrections were made is proof of their practical utility. Recurrence of the same assignable cause, for example, scale errors, shows that the assignable causes were not permanently removed. Although permanent elimination is the goal, a recurrence is not the fault of the chart. Instead, the chart helps to identify the offender. Management will wisely take notice and compare the cost of a new batch-scale installation with the cost of poor quality production or the cost of excessive cullet.

### (2) New Central Line after Batch Change

In the usual applications of control charts, it is customary to compute a new central line from new data representing 20 or more subgroup samples or 60 or more individuals. However, in applying control charts to glass density, when the change is a deliberate batch change, it is possible to predict the new central line accurately in advance.† The known composition change on an oxide basis is converted to a density difference, and this difference is applied to the old central line. Examples of deliberate batch changes are shown in Fig. 9; another example is furnace A, Fig. 3.

When starting a control chart, if the mistake of extending a central line at an improper level is made, revisions may be made as they become necessary.

## VI. Keeping Lack of Control Within Tolerable Limits

### (1) Tolerable Changes in Density Level‡; Composition Equivalents

The present application of control charts differs from most applications because the absolute value of the central line density is not a critical quality characteristic of commercial glass. It is unlike the finish dimension of a jar: If the diameter is not $2 \pm 0.020$ in. or some such value, the cap will not fit. In the case of glass density, the important thing is not the average density over a long period but abrupt changes in the density level; different levels require different fabrication techniques on the forming machines. If the level were maintained constant over a long period, the workmen would be able to fabricate good quality ware out of glass of any reasonable density, but they cannot quickly accommodate their technique to abrupt changes. Thus it is necessary to set some limits on the density level or at least to make only long-term changes.

A gradual change in the level, provided the range

---

* The control chart does not locate the assignable causes; it merely indicates when they exist. The plant technician has to find them by going to work in the plant and laboratory, using his best hunch as a starting point and keeping in mind that the subgroup sample being used was chosen to show day-to-day variations in the batch house.

† See reference (2), Table IV, p. 263; an illustration of the computation of density difference from a known composition change, $\Delta X$, is given herewith:

| Oxide | $\Delta X$ | Factor, $Y_M$ | $Y_M \Delta X$ |
|---|---|---|---|
| $SiO_2$ | 0.30 | −0.0273 | −0.0082 |
| $Al_2O_3$ | 0.20 | −0.0232 | −0.0046 |
| CaO | −0.30 | −0.0143 | 0.0043 |
| $Na_2O$ | −0.10 | −0.0200 | 0.0020 |
| BaO | −0.10 | −0.0075 | 0.0007 |
| | 0.00 | | −0.0058 |

The calculated density change is −0.0058.

‡ The term "density level" as used here means the average of daily density values taken over a period of several days, or a few weeks, during which the density values show no particular upward or downward trend.

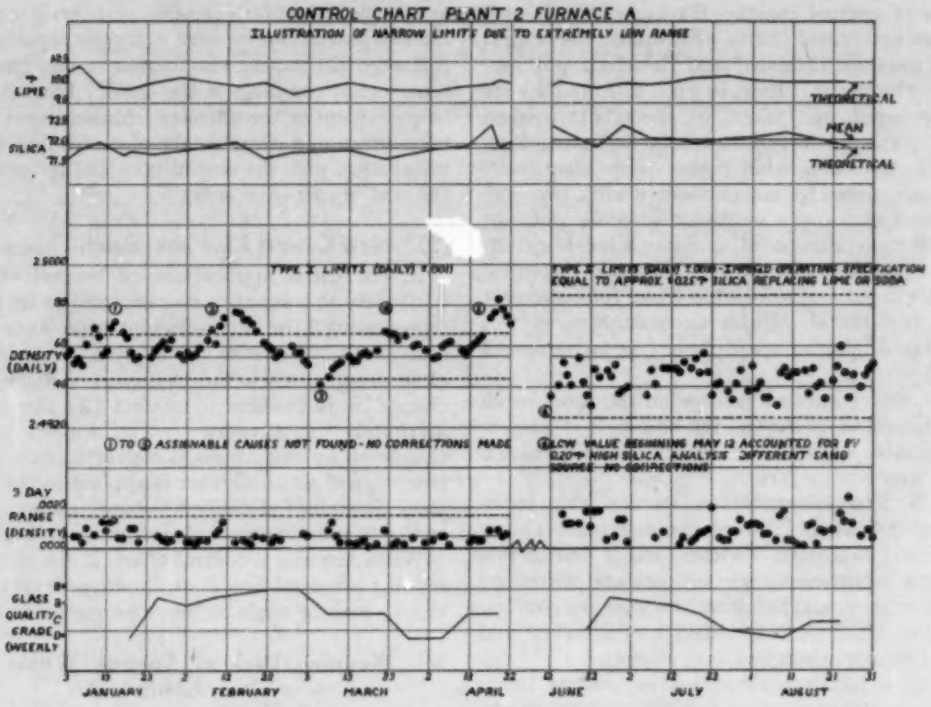

CONTROL CHART PLANT 2 FURNACE A
ILLUSTRATION OF NARROW LIMITS DUE TO EXTREMELY LOW RANGE
LIME
SILICA
THEORETICAL MEAN
THEORETICAL
TYPE I LIMITS (DAILY) ±.0011
TYPE II LIMITS (DAILY) ±.0030 - ENFORCED OPERATING SPECIFICATION EQUAL TO APPROX. ±.025% SILICA REPLACING LIME OR SODA
DENSITY (DAILY)
① TO ⑤ ASSIGNABLE CAUSES NOT FOUND - NO CORRECTIONS MADE
LOW VALUE BEGINNING MAY BE ACCOUNTED FOR BY 0.20% HIGH SILICA ANALYSIS DIFFERENT SAND SOURCE NO CORRECTIONS
3 DAY RANGE (DENSITY)
GLASS QUALITY GRADE (WEEKLY)
JANUARY
FEBRUARY
MARCH
APRIL
JUNE
JULY
AUGUST

FIG. 10.—The 3-sigma limits and central line were established for a previous period and extended on this chart. Assignable causes 1 to 5 are noted but they were neither accounted for nor corrected. The evident change in level beginning June 12 is accounted for and represents a 0.20% increase in $SiO_2$ due to a new source of the sand. The 3-day range in the period June 12 to August 31 appears to be greater than that of the previous period. If this situation continues through September 15, not shown, a new $\bar{R}$ should be calculated and the corresponding control limits used. If the new limits for density are less than $\pm 0.0030$, then type II, or modified, limits of $\pm 0.0030$ may be used anyway.

does not go out of control, will not cause cord or upset production (see Fig. 9, period July 15 to July 31). Experience has also established the fact that a batch change and the accompanying change of density level can be made without causing difficulty, provided it is made slowly. Using the control chart technique, the change is made in such a way that the density range stays within its statistical control limits; it is guesswork no longer.

In Fig. 10 a lack of control of density is indicated, although the range is under control most of the period. Actually the change is small and the glass quality was satisfactory ($D+$ glass quality corresponds with $C+$ ware grade). The assignable causes on the density charts correspond to a variation of the density of about $\pm 0.0025$ from the central line. This is equivalent to approximately $\mp 0.20\%$ replacement of lime by silica. In the present state of the art, most laboratories would consider this a tolerable variation and would not consider it worth while to find and eliminate such assignable causes. Of course they could be found because the process has set its own narrow limits, and the fact that it is out of control indicates that there is something unusual.

### (2) Modified Control Limits

Under the above circumstances, it is suggested that an imposed operating specification limit of $\pm 0.0030$ could be used for the central-line density, and variations within this band not taken seriously, at least in the present state of the art. This would not be done, however, if one is seeking to improve the process.

This $\pm 0.0030$ band corresponds to a $\mp 0.25\%$ composition change of $SiO_2$ replacing CaO, a change in composition that is not considered critical. Such modified, or type II, limits are shown in Fig. 10.

In Fig. 5 are shown similar operating specification limits for individuals, and also for the corresponding subgroup averages. The spread for the density is $\pm 0.0050$, corresponding to $\mp 0.40\%$ $SiO_2$ replacing CaO. But since $\mp 0.40\%$ is probably too large a composition change for satisfactory operation, it is recommended that the statistical, type I, limits be retained instead. In fact, trouble was experienced with checks in the ware in the period June to July. This confirms the belief that it is not desirable to allow a variation in level much greater than the $\pm 0.0027$ statistical band. Modified limits greater than $\pm 0.0030$ are therefore not recommended for density. Even when limits of $\pm 0.0030$ are used for the density, it is essential that the range of density stay under strict statistical control.

## VII. Discussion

### (1) Rational Subgroups; Other Methods of Subgrouping

The recommended method as described involves the

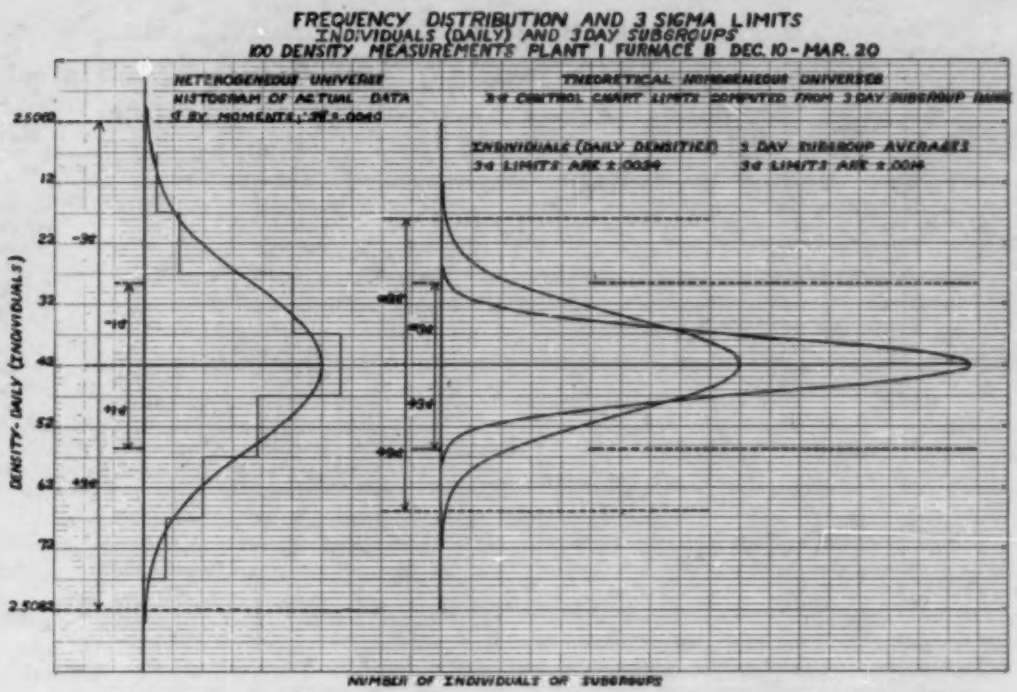



FIG. 11.—Normal frequency distribution curves are plotted with frequency as the abscissa in order to illustrate the derivation of 3-sigma limits used on control charts. The left-hand histogram and curve illustrates 3-sigma limits that are too wide, the value of sigma computed from moments being too large because of trends in the level of the 100 consecutive daily densities. For the two curves on the right, 3-sigma limits derived from 3-day range of density are indicated. They illustrate the relation between 3-sigma limits for individuals and for subgroup sample averages.

use of three consecutive daily density values from a particular furnace as a subgroup sample. It is possible, then, to know in advance, when trouble occurs, that the cause will most likely be found in the batch house.

Two other methods are illustrated in Table II, columns (6) and (7).. One of them, a subgroup sample of two consecutive days on three furnaces ($n = 6$), is unsatisfactory because it includes furnace differences as well as day-to-day batch-house variation. Thus the sample is stratified (not homogeneous). The other method of taking three furnaces on the same batch house each day as a sample ($n = 3$) is a test of the variability among furnaces. This might be useful but the result would be obscured by the batch-house variation when it is the dominant factor, as in the examples given. A chart on both could be run, however, that is, one on $n = 3$ days and one on $n = 3$ furnaces. In the latter chart, it would be necessary to go back to the furnaces to look for trouble, if any occurred.

If the variability of the glass flow or other characteristics of a particular furnace is to be tested, the rational subgroup would be the densities of all shops taken simultaneously ($n$ = the number of shops).

There are, of course, other possible subgroups that would show essentially day-to-day variations in the batch house; for example, 3 densities taken every other day from the same furnace, 4 densities taken morning and night on two consecutive days, and 3 or 4 densities taken for the various shifts of a particular day. The difficulties with these samples are that the intervals represented by the samples are either too long or too short to show up the fluctuations characteristic of the batch house, if the furnace back log of glass is 50 to 150 tons. Although more work should be done on the effect of sample interval, it is believed that three consecutive daily densities is the most satisfactory sample for preliminary applications of control charts.

## (2) Range of Density

Referring again to the line of best fit in Fig. 1, it is seen that if one were to impose an operating specification on density range such that a cord grading lower than C+ would not be produced, the 3-day density range specification limit would have to be not greater than 0.0030.* This would correspond to the upper control limit of the range chart. The average range would then have to be approximately 0.0012, (0.0030/2.574), and the width of the daily density chart limits ±0.0020, (±3 × 0.0012/1.693).

It is interesting to compare the actual ranges and limits for all furnaces for which data are given. This is summarized in Table III, columns (1) to (5). More than one half of the furnaces and plants fall within an average 3-day range of 0.0013. Plant 5 was deliberately selected as an example of high range. The goal of all plants should be a 3-day range of density of 0.0012 or less.

* This conclusion is based on the line of best fit for one furnace only; it is not intended to be a final statement of fact but merely an illustration of the method.

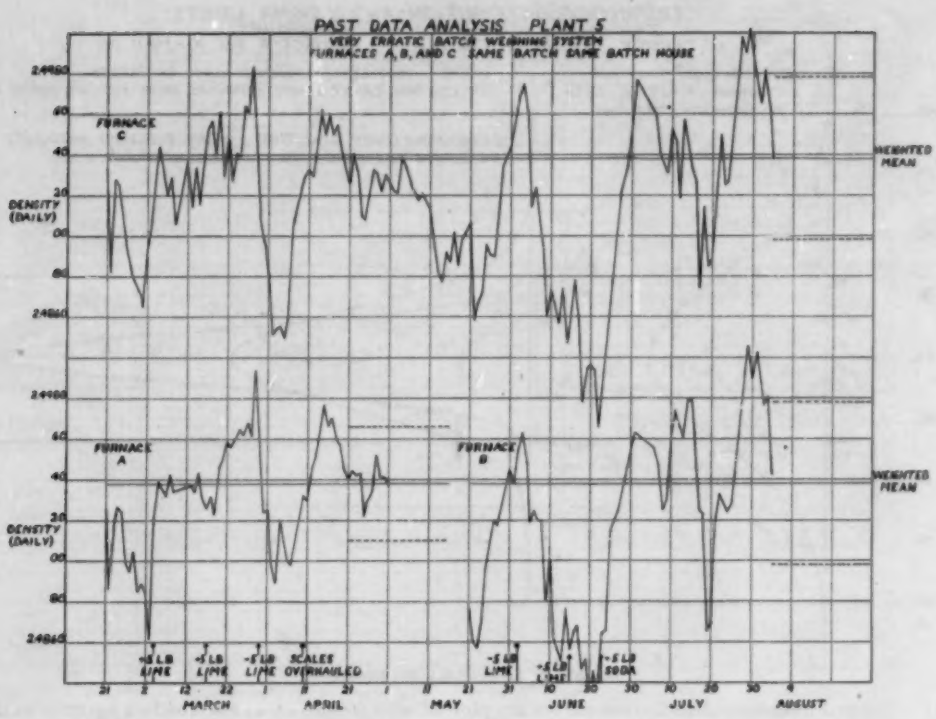



FIG. 12.—Daily density curves for three furnaces in a plant where the poor batch system produced a very wide density range. The limits for the erratic batch weighing system are indicated at the margins of the curves. All curves have the same central-line density of 2.4938, but the density for the largest furnace, furnace A, has the least variability and hence the narrowest limits. Batch changes, which were made in order to correct for scale errors, are shown at the bottom of the chart.

## (3) Statistical Character of Density Data

Reference is made to Figs. 6 and 7, dates December 10 to March 20. The average density during this 100-day period was 2.4942. This is a period of past data analysis and the limits have been put on the chart in retrospect (as is the case of all charts ahead of the period labeled "extension"). Note the evidence of lack of control and the evidence of trends in the density. In other words, the daily densities constitute a heterogeneous universe, or rather a mixture of universes; conditions are changing throughout the period. The value of sigma computed by the regular moment method is 0.0013. The frequency distribution, or histogram, along with the theoretical normal curve for $n = 100$, sigma = 0.0013, is shown on the left hand side of Fig. 11. The 3-sigma band is also indicated. But as already indicated above, this is not the proper 3-sigma band because of trends and lack of control in the 100 consecutive daily density values. The variability, however, was under control during this period, as indicated by the range chart for the period. Therefore the best measure of sigma for the process is the one derived from range. This value is 0.0008. On the right-hand portion of the figure is shown the corresponding 3-sigma limits and the corresponding normal frequency distribution curves for both individuals and subgroup averages.

## (4) Density vs. Chemical Analysis as a Method of Control

In general, a change of 0.001 in density indicates an approximate 0.1% change in any one constituent.* Now the standard deviation of the plant laboratory measurement of density on a particular sample is probably about 0.0002, while for chemical analysis it is probably about 0.10 to 0.20% for some constituents. Thus it is obvious that density is the more sensitive method. Another way of looking at this is to compare the density control limits of $\pm 0.0020$ to $\pm 0.0030$ for most furnaces with the equivalent oxide limits of $\pm 0.15$ to $\pm 0.25\%$. It is seen that the control-chart limits for density, which include measurement errors and all other variations in the process, are not much wider than the probable error of a chemical analysis in a plant laboratory on a particular sample.

In Fig. 7, on the daily density curve for the period June to July, the crosses indicate densities computed from chemical analyses. The spread is much wider than that of the actual, measured density. Since density difference factors were used, and the use of absolute values thereby avoided, this discrepancy cannot be accounted for by errors in the factors. The

* See reference (1) p. 386; these relationships for various oxide additions or replacements can easily be derived from the $Y_M$ factors given in Table IV, p. 263, of reference (2).

TABLE III

DENSITY VARIABILITY IN SIX PLANTS AND TEN FURNACES AS MEASURED BY 3-DAY RANGE OF DENSITY

| (1) Plant and furnace | (2) Fig. No. | (3) Periods | (4) Avg. 3-day range ($\bar{R}$) | (5)* Width of limits | (6)† Approx. $SiO_2$ equivalent | (7) Cord quality | (8)‡ Relative size | (9) Batch-house equipment |
|---|---|---|---|---|---|---|---|---|
| 2A | 10 | July–Dec. | 0.0006 | ±0.0011 | ±0.09% | Good | Large | Good |
| 3A | 8 | Sept.–March | 12 | 21 | .19 | Fair | Large | Modern |
| 3A | 9 | April–June | 9 | 16 | .13 | Good | Large | Modern |
| 6A | | Jan.–April | 12 | 21 | .19 | Good | Large | Modern |
| 1B | 6 and 7 | Dec.–June | 13 | 22 | .20 | Poor | Large | Poor |
| 1A | 4 and 5 | Jan.–June | 15 | 27 | .21 | Fair | Small | Poor |
| 4A | 2 and 3 | April–Aug. | 13 | 22 | .20 | Fair | Large | Fair |
| 4B | 2 and 3 | " " | 16 | 28 | .22 | Fair | Small | Fair |
| 5A | 12 | Feb.–May | 18 | 32 | .25 | Poor | Large | Poor |
| 5B | 12 | " " | 23 | 40 | .31 | Poor | Medium | Poor |
| 5C | 12 | " " | 23 | 40 | .31 | Poor | Small | Poor |

* Daily density measurements (individuals).

† 0.10% $SiO_2$ replacing CaO is approx. 0.0013 decrease in density. This is a maximum value; other composition changes or replacements give somewhat less density change.

‡ As compared with other furnaces on same batch plant.

method is as follows: Compare the analytical report with theoretical analysis and determine the composition difference; then use the density difference factors, to which reference has already been made, for computing a density difference from the composition difference, and apply this difference to the central line density.* Between June 19 and June 26, the actual density increased from 2.5006 to 2.5024, while the computed density decreased from 2.4988 to 2.4960. This illustrates the difficulties in using chemical analysis as a method of control. Its best use would seem to be as a trouble-shooting tool to locate assignable causes when they occur on a density control chart. Even here, however, present experience indicates that the first thing to do is to go to the batch house and check the scales.

### (5) Comments on Utility and Limitations of Control Charts

In Fig. 12 is an example of a situation where something more fundamenral than a chart is needed. The limits on the right-hand margins of the curves are representative of the typical variability after throwing out a lot of obvious mavericks. It would do no harm to use the limits and try to establish control but it probably wouldn't do much good either. Assignable causes are so obvious, and occur so frequently, and trends are so pronounced, that the yardstick of 3-sigma limits yields little additional information. This argument might be applied to all charts, but the author's opinion, supported by the experience of those who used the charts shown, is that the 3-sigma criteria are well worth the simple arithmetical work necessary to install them.

The time required for past data analysis is about one day for each furnace. The plotting of points on a current chart used for control purposes requires about one minute each day; and notation, adjustment, and review require about one day each month. The simple daily density chart as used in many laboratories can easily be made into a control chart; it is merely a matter of adding a range chart and inserting central lines and limits. The use of the composition and ware-grade charts is not an essential part of the density-control method; they are included merely as an aid in studying related quality characteristics. Some plant laboratories plot the composition of all ingredients. For simplicity, only silica and lime have been shown in the figures in this paper.

Present experience indicates that control charts are a practical and valuable aid to the plant and management.

## VIII. Summary

(1) Statistical control charts are practical, and can be applied advantageously in glass plants to glass density data for (*a*) analysis of causes of variation, (*b*) maintaining a state of statistical control during current production, and (*c*) keeping lack of control within tolerable limits.

(2) The dominant variability and the cause of many unusual variations in density lie in the batch house—often in the scales.

(3) A subgroup sample consisting of three consecutive daily densities from a particular furnace is the most useful sample for control purposes.

(4) An average 3-day range of density of not more than 0.0012 is a reasonable goal for any plant; 0.0006 is excellent and 0.0009 is good performance.

(5) The average range of 3-day subgroup samples is a powerful aid in studying related quality characteristics.

(6) Chemical analyses are usually less precise than density measurements and can best be used only for trouble-shooting purposes.

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*In using density difference factors for computing a density difference, care has to be taken at the start that the algebraic sum of all composition differences is zero. It will be zero, of course, if both the theoretical composition and the laboratory report on the sample at hand total 100.00%. Many laboratory reports, however, total 99.81% or some such figure. This error alone can give a density discrepancy wider than the control limits. Under these circumstances, it is known immediately that the analysis is wrong or at least is merely an approximation. In using this kind of laboratory report for density calculations (a doubtful procedure), it is necessary to adjust the total to 100.00%, making the adjustment in $SiO_2$, unless there is good reason to make it elsewhere.

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# INDEXES TO THE JOURNAL

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## AUTHOR INDEX

The reference number in parentheses refers to the *monthly* issue of the *Journal;* the numbers following are the *page* numbers.


**Allen, Dan.** See Galbreath, W. W., Jr.; McVay, T. N.
**Amberg, C. R.** Effect of additions of diopside on properties of wall-tile body, (11) 324–26.
**Andrews, A. I.** See Blanchard, M. K.; Fréchette, V. D.; Petersen, F. A.
**Andrews, A. I., and Swift, H. R.** Effect of composition and temperature on solubility of iron oxide in ground-coat enamel glass, (2) 46–50.

**Baumann, H. N., Jr.** Fusion experiments in system MgO–FeO–$Cr_2O_3$–$Al_2O_3$, (11) 327–29.
**Beals, M. D., and Zerfoss, Samuel.** Volume change attending low-to-high inversion of cristobalite, (10) 285–92.
**Birch, R. E., and Hall, M. P.** Some factors affecting P. C. E. of silica fire clay, (8) 239–41.
**Bishop, F. L., Jr.** Surface layer of sheet glass, (5) 145–48.
**Blanchard, M. K., and Andrews, A. I.** Enamel film strength as affected by properties of enamel before, during, and after drying, (1) 25–31; Fundamental properties of soluble salts in enamel mill liquors, (1) 17–24.
**Blanchard, M. K.** See Harman, C. G.
**Blount, E. B.** See Pence, F. K.
**Brookover, George.** See Potts, J. C.
**Burch, O. G.** See Potts, J. C.
**Burnham, Forrest, and Harder, C. M.** Practical production of one-fire luster glazed pottery, (2) 62–64.

**Carter, H. D.** See King, B. W., Jr.
**Cohn, Gunther.** Significance of topochemical factors on rate of reactions between solid salts, (10) 308–16.
**Coughanour, W. L., and Utter, J. L.** Cataphoresis of purified fractionated kaolinite particles, (4) 116–20.

**Draker, H. C.** See King, B. W., Jr.

**Fabianic, W. L.** Glasshouse stones, (11) 330–50.
**Falcão, E. de R.** See Scholes, S. R.
**Fraser, W. A., and Upton, L. O.** Optical fluor-crown glasses, (4) 121–28.
**Fréchette, V. D., and Andrews, A. I.** Investigation of reactions of simple magnesia spinels with alkaline earth orthosilicates in solid state, (7) 197–202.

**Galbreath, W. W., Jr.** See McVay, T. N.
**Galbreath, W. W., Jr., McVay, T. N., and Allen, Dan.** Lightweight superduty kyanite-topaz refractory, (8) 241–46.
**Ghering, L. G.** Refined method of control of cordiness and workability of glass during production, statistical control charts applied to daily measurements of density, (12) 373–87.
**Ghering, L. G., and Knight, M. A.** Properties and diagnosis in pressed and blown glassware, (9) 260–66.
**Glass Container Association Ring Section Sub-Committee of Standard Testing Procedure Committee.** Polariscopic examination of glass-container sections, (3) 85–89.
**Goudge, M. F.** Brucite magnesia, (1) 8–10.
**Grim, R. E., and Rowland, R. A.** Differential thermal analysis of clays and shales, and control and prospecting method, (3) 65–76.

**Hall, M. P.** See Birch, R. E.
**Harder, C. M.** See Burnham, Forrest.
**Harman, C. G.** Suggestions for solution of difficult glaze-fit problem, (8) 231–33.
**Harman, C. G., and Blanchard, M. K.** Study of factors involved in glaze-slip control, correlation of glaze thickness with air permeability, II, (7) 207–209.
**Harman, C. G., and Johnson, H. C.** Study of factors involved in glaze-slip control, additional glazes and improved technique and apparatus, III, (7) 209–14; practical uses for coherence value and receptivity measurements, IV, (7) 214–20.
**Harman, C. G., and Schaefer, C. F.** Study of factors involved in glaze-slip control, glaze-slip specifications, I, (7) 202–207.
**Hausner, H. H.** Influence of humidity on dielectric properties of high-frequency ceramics, (6) 175–81.
**Herman, A., and Shay, H. L.** Effect of glass on alcoholic products stored in bottles at room temperature for three years, (2) 53–57; correction, (12) 401.
**Huggins, M. L., and Sun, K.-H.** Changes of properties of glass resulting from changes of composition, (1) 10–12; Glass properties and the periodic table, (1) 13–17.
**Hunt, H. J.** Analysis of batch for segregation, (10) 305–308; see also Zarbo, G. J.
**Hutchison, Clark.** Abrasion resistance of porcelain enamels, (12) 366–69.

**Johnson, A. G., and Underhill, D. B.** Quantitative determination of the glassy phase in a ceramic body, (2) 40–41.
**Johnson, A. L.** See Norton, F. H.
**Johnson, H. C.** See Harman, C. G.

**King, B. W., Jr., Carter, H. D., and Drake, H. C.** Properties of enamel slips, general properties of clays and enamel slips, I, (9) 253–60.
**King, R. M.** Mechanics of enamel adherence, influence of manganese dioxide on metal precipitation at ground coat-iron interface, XVI, (11) 350–51.
**Kingslake, R.** Optical glass from viewpoint of lens designer, (6) 189–95.
**Knight, M. A.** Surface tension and persistence of cords in glass, (5) 143–45; see also Ghering, L. G.

**Lamar, R. S.** Particle shape and differential shrinkage of steatite talc bodies, (11) 317–20.
**Lauck, F. W.** See Martin, W. G.
**Lawrence, W. G.** See Norton, F. H.
**Lyon, K. C.** Calculation of surface tension of glasses, (6) 186–89.

**Marquis, John.** Cone 01 glazes for low-temperature vitreous ware, (12) 358–66.
**Martin, W. G., and Lauck, F. W.** Observations indicating absence of plastic flow in glass coating on steel, (11) 352–54.
**McCutchen, E. S.** Strontia and its properties in glazes, (8) 233–38.
**McDevit, W. F.** Effect of firing atmosphere on development of colors in ceramic glazes, (6) 165–75.
**McVay, T. N.** See Galbreath, W. W., Jr.
**McVay, T. N., Galbreath, W. W., Jr., and Allen, Dan.** Substitution of topaz, domestic kyanite, and synthetic corundum for India kyanite, raw topaz as high-temperature bond for domestic kyanite, IV, (9) 275–82.
**Mohr, W. C.** See Russell, Ralston, Jr.

**Nordberg, M. E.** Properties of some Vycor-brand glasses, (10) 299–305.
**Norton, F. H., and Johnson, A. L.** Fundamental study of clay, nature of water film in plastic clay, V, (3) 77–80.
**Norton, F. H., Johnson, A. L., and Lawrence, W. G.** Fundamental study of clay, flow properties of kaolinite-water suspensions, VI, (5) 149–64.
**Noyes, Bradford, Jr.** See Taylor, N. W.

**Owens-Illinois Glass Company General Research Laboratory.** Effect of fluorine and phosphorus pentoxide on properties of soda-dolomite lime-silica glass, (12) 369–72; Effect of substituting MgO for CaO on properties of typical soda-lime glasses, (8) 221–25.

**Pence, F. K., and Blount, E. B.** Vermiculite as raw material in ceramic manufacture, (2) 50–52.
**Petersen, F. A., and Andrews, A. I.** Enamel Utensil Manufacturers' Council tentative standards solubility test for porcelain enameled cooking utensils, (4) 113–15.
**Peterson, C. A.** See Robinson, H. A.
**Pole, G. R.** Calcination of topaz ore in rotary kiln, (6) 181–85.
**Potts, J. C., Brookover, George, and Burch, O. G.** Melting rate of soda-lime glasses as influenced by grain sizes of raw materials and additions of cullet, (8) 225–31.

**Robinson, H. A., and Peterson, C. A.** Viscosity of recent container glass, (5) 129–38.
**Rowland, R. A.** See Grim, R. E.

**Russell, Ralston, Jr., and Mohr, W. C.** Casting characteristics of clays, improved methods for determination, I, (4) 97–113.
**Russell, Ralston, Jr., and Weisz, R. S.** Some particulate characteristics of commercial flint and feldspar, (1) 1–7.

**Schaefer, C. F.** See Harman, C. G.
**Scholes, S. R., and Falcão, E. de R.** Rapid method for estimating soda in glasses, II, (5) 148.
**Schramm, Edward.** Glost kiln refractories, (9) 282–84.
**Seabright, E. C.** Methods for testing cleaner solutions, (3) 90–95.
**Shay, H. L.** See Herman, A.
**Silverman, W. B.** See White, J. F.
**Smith, D. C.** See Zarbo, G. J.
**Sun, K.-H.** See Huggins, M. L.
**Swift, H. R.** See Andrews, A. I.

**Taylor, N. W., and Noyes, Bradford, Jr.** Aging thermometers, (2) 57–62.
**Thiemecke, H.** Notes on moisture expansion, (12) 355–58.
**Tiede, R. L.** See Tooley, F. V.
**Tooley, F. V., and Tiede, R. L.** Factors affecting degree of homogeneity of glass, (2) 42–45.
**Tracey, J. I., Jr.** High-alumina clays of Pulaski and Saline counties, Ark., (5) 246–49.

**Ueltz, H. F. G.** Crystalline and glassy phases of steatite ceramics, (2) 33–39.
**Underhill, D. B.** See Johnson, A. G.
**Upton, L. O.** See Fraser, W. A.

**Utter, J. L.** See Coughanour, W. L.

**Weisz, R. S.** See Russell, R., Jr.
**White, J. F., and Silverman, W. B.** Devitrification characteristics of alkali-lead oxide-dolomite lime-silica glasses, (3) 81–85.
**White, J. S.** Particle-size distribution of steatite talc, (11) 320–23; for correction see (12) 401.
**Winter, Aniuta.** Time problem in annealing (9) 266–74.

**Zapffe, C. A.** Conversion of certain refractory oxides to a suboxide form at high temperatures, (10) 293–98.
**Zarbo, G. J., Hunt, H. J., and Smith, D. C.** Analysis of cords and stones, (5) 138–42.
**Zerfoss, Samuel.** See Beals, M. D.

---

# SUBJECT INDEX TO THE JOURNAL

## Volume 27, 1944

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**Abbe values** for pure oxides, curves and equation for calculation, (1) 15–16.
**Abramson formula for particle concentration,** (4) 117.
**Abrasion resistance of porcelain enamels,** (12) 366.
**Achromatism** in optical glass, description, proportionality, and diagram of method, (6) 189–91.
**Acid solubility, test for enameled utensils,** tentative standard test of E. U. M. C., (4) 113.
**Acid tests** for luster glazes, (2) 64.
**Adams and Williamson equation** for relation of reciprocal of stress with time, (2) 58.
**Air-separator method.** See *Methods.*
**Alcoholic products,** hydrogen-ion concentration of, effect of commercial bottles after storage for three years at room temperature, (2) 53.
**Alkaline earth orthosilicates** in solid state, reaction of simple magnesia spinels with, (7) 197–202; optical properties, (7) 199.
**Alkalinity** of cleaner solutions, equations for calculation, (3) 91.
**Alumina and silica** in glass, semimicro-determination method for analysis, tables, (5) 139–41.
**Alumina clays,** high-alumina clays in Ark., deposits, chemical analyses, geologic history, and availability, (8) 246–49.
**American Society for Testing Materials,** standard method, mechanical analysis of soils. See *Methods, hydrometer.*
**Anastigmat lenses,** design, (6) 194; see also *Glass, optical.*
**Andreasen pipette method.** See *Methods.*
**Annealing** of glass. See *Glass.*
**Apparatus,** air separator, laboratory type, for particle-size measurements, (11) 320.
for atmosphere control in furnaces for ceramic glazes, (6) 166–67.
Babinet compensator for measuring stresses in glass, (3) 85.
for $BaSO_4$ and quinolate filtration and micro-oven assembly, (5) 151.
dilatometer for volume-expansion tests on cristobalite, (10) 287.
drier and stirrer for clays for slip preparation, (4) 99.
for enamels, for solubility tests on enameled utensils, (4) 114–15.
furnaces. See *Furnaces.*
for glass, stones and cords in, semimicro-determination, (5) 141.
for glass surface-tension measurements, diagram, photo, (5) 145–46.
for glaze-coherence tests, diagram, (7) 204; for measuring coherence values and body receptivity, photo, (7) 210.
for glaze slips for surface-tension measurements, diagram, (7) 204.
for glaze-thickness tests, mechanical dipper, (7) 208.
for glazes, constant heating schedules, (6) 167.
homogeneity equipment for tests on soda-lime-silica glass, photo, (2) 42; centrifuge head, diagram, (2) 43.
**Apparatus,** (*continued*)
hydrometers. See *Hydrometers.*
MacMichael viscosimeter, modified, for data on kaolinite-water suspensions, (5) 158–63.
microscope, polarizing, for examination of glass-container ring sections, (3) 88.
for particle-size measurements, (11) 320–21.
for permeability, casting rate, and water retention of clays, (4) 99–100.
for semimicro-determination of stones and cords in glass, (5) 141.
Stormer viscosimeter for thixotropy tests, (4) 99.
viscometers, viscosimeters. See *Viscosimeters.*
for volume-expansion tests on cristobalite, (10) 287; expansion bulb and thermocouple, photo, diagram of furnace and expansion bulb, (10) 288.
for water-film thickness of plastic clays, mold cross section, (3) 78.
**Arkansas clays.** See *Clays, high-alumina.*
**Arrhenius exponential law** for viscosity of suspensions vs. concentration, (5) 151.
**Autoclaves** for chemical durability of glass bottles, (2) 54.
steam tests on talc-pyrophyllite bodies, tables, (12) 356.
test results on glazes for low-temperature vitreous ware, (12) 364.

**Balinkin equation** for evaluation of small overall color differences, (6) 166.
**Barium chloride–$H_2O$,** equilibrium system, diagram, (1) 21; $BaCl_2$–$MgCl_2$, equilibrium system, diagram, (1) 23.
**Barium fluoride–$BaCl_2$,** equilibrium system, diagram, (1) 23.
**Barium orthosilicate,** synthesis, tables, and reaction test, (7) 200–201.
**Batch, glass.** See *Glass, batch.*
**Batch stones** in glass. See *Glass, cords and stones; Stones.*
**Bausch and Lomb Optical Co.,** optical glass types and characteristics, (6) 190.
**Bibliography** of chemical reactions, (1) 23–24.
of equilibrium systems, (1) 24.
glass, devitrification and stone developments, (11) 350.
**Bisque bodies** and glazed semivitreous ware, moisture-expansion studies, curves, (12) 355–57.
**Blisters** and coloring material in bottle glass, source, detection, photomicrograph, (11) 348.
**Bottles, glass.** See *Glass, bottles; Stones.*
**Brewsterian angle,** apparent, Pfund method for determination, (5) 145.
**Brucite magnesia,** sources, characteristics of granular product, uses, (1) 8–10.
**Brucitic limestone,** description and processing, (1) 8.
**Büchner funnel** vs. cup-casting method for casting slip, (4) 111.

**Calcination** of topaz ore in rotary kiln, (6) 181–85.
**Calcium chloride–$H_2O$,** equilibrium system, diagram, (1) 20.
**Calcium chromate,** thermal decomposition, reactions, (6) 169.
**Carman's permeability method,** rate of flow, (11) 319.
**Casagrande hydrometer method,** (11) 320.
**Casting characteristics** of clays, improved methods, (4) 97; see also *Slips.*
**Cataphoresis** of purified, fractionated kaolinite particles, ultramicroscopic method, (4) 116.
**Ceramic ware,** glassy phase, method of determination, (2) 40.
**Charts,** statistical control, for daily density measurements of glass, (12) 373–87.
**Chemical reactions.** See *Reactions.*
**Chinaware,** hotel, and semivitreous dinnerware, body composition, glaze coherence, and receptivity test data, (7) 211–13; see also *Dinnerware; Vitreous ware.*
**Chrome-corundum,** PeO and MgO additions, effect of $Cr_2O_3$–$Al_2O_3$ content, curves, (11) 329; effect of varying $Cr_2O_3$ and MgO fused with $Al_2O_3$, (11) 329.
fusion studies, effect of varying MgO and $SiO_2$, curves, (11) 328; effect of adding $TiO_2$, (11) 328.
**Chrome-lime,** mixtures, colors when calcined at various atmospheres, diagram, (6) 168.
**Chrome ore,** mineral composition, (11) 327.
**Chrome refractories,** effect of chemical and thermal treatment, (11) 327.
**Chrome–tin system,** colors developed in, (6) 174.
**Chromium oxide–$Fe_2O_3$,** X-ray studies, (6) 173.
**Clay plants,** operating pits, thermal analyses of clays, control method, (3) 65.
**Clay stones.** See *Stones.*
**Clays.** See also *Kaolin.*
ball, for slip casting, effect of electrolyte content, curves and data, (4) 109–11.
casting characteristics, improved methods, (4) 97.
clay-flint-feldspar bodies, moisture-expansion data, (12) 357.
clay minerals, kaolinite, hydrated halloysite, illite, and bentonite, thermal reactions, curves, (3) 67.
clay minerals, pure forms and synthetic mixtures, method for differential thermal analyses, (3) 65.
clay suspensions, flow properties of kaolinite-water suspensions, (5) 149.
clay suspensions, for study of cataphoretic velocity of purified fractionated kaolinite particles, (4) 116.
for common brick, thermal curves, (3) 75.
for enamel slips, physical and chemical properties, (9) 253–54; carbon content and opacity data, (9) 257.
in enamels, fired, properties, (9) 257–58.
fundamental studies, nature of water film in plastic clays, V, (3) 77; flow properties of kaolinite-water suspensions, VI, (5) 149.

**Clays** (*continued*)
Georgia, thermal curves for bauxitic kaolin, soft, hard, and plastic kaolins, and fuller's earth, (3) 71–73.
German glasspot, thermal curves, (3) 73.
gibbsite, boehmite, diaspore, quartz, calcite, dolomite, and gypsum, thermal reactions, curves, (3) 68.
for glazes, base-exchange capacity vs. coherence value, curve, (7) 219.
high-alumina, Ark., location and description of deposits, (8) 246; chemical analyses, geologic history, and availability, (8) 247–49.
hydrogen-ion vs. NaOH concentration, curves, (9) 254.
Illinois, thermal curves for kaolin and fire clays, (3) 71, (3) 73–74.
Missouri burley flint and plastic fire clay, thermal curves, (3) 71, (3) 73.
natural, thermal curves, (3) 71.
Ohio, thermal curves for flint, ball, and fire clays, (3) 71, (3) 73.
organic materials and minerals in, clarain, vitrain, limonite, marcasite, pyrite, and goethite, thermal reactions, curves, (3) 69.
permeability, casting rate, and water retention, test apparatus, (4) 99–100.
settling tests, maximum particle-diameter data, (9) 255.
and shales, differential thermal analysis, (3) 65; thermal curves, vertical scales for temperature-difference data, (3) 67, see also *Shales*.
for slips, drier for moisture compensation and stirrer, (4) 99.
for slips, enamel, general properties, (9) 253.
in slips, single clays and in combination, properties, (9) 258–60.
synthetic mixtures, thermal curves, (3) 70.
Tennessee ball, for glaze slips, coherence and surface-tension values, curves, (7) 205–206.
Tennessee, sagger and wad, thermal curves, (3) 72.
Texas, for glaze slips, coherence and surface-tension value curves, (7) 205–206.
water film in, simple clay–water system, water-film thickness and stability tests, (3) 77–79.
X-ray patterns, (9) 254.
**Clayware**, clay-vermiculite bodies, effect of temperature on properties, thermal-expansion data at cone 08, tables, (2) 52.
dust-pressed and plastic processes for bodies with vermiculite as raw material, properties and comparison of bodies, table, (2) 50–51.
structural, thermal-conductivity and sound-absorption measurements, effect of vermiculite in, (2) 50–51.
**Cleaner solutions**, "activity factor" or "available alkali" formulas, (3) 90; available alkali, calculated percentage daily results, (3) 92.
alkalinity and sodium hydroxide content, formulas for calculation, (3) 91–93.
for enamel industry, test methods, soap test and free alkali and soluble soap test, (3) 90–91; sheet cleaning, cleaner strength, available alkalinity and soap and free-alkali tests, (3) 93–95.
**Cobalt oxide** with $Ni_2O_3$ and $MnO_2$ in enamels, effect on metal precipitation at ground coat-iron interface, (11) 350–51.
**Coherence values** for slip glazes. See *Glazes; Slips*.
**Colors**, brown, composition and reflectance measurements, (6) 173–74.
in ceramic glazes, effect of firing atmosphere, (6) 165.
color equivalents in flint glass, with and without tint plate, (3) 87.
designation, hue, lightness, saturation, definitions, (6) 165.
of glazes, measurement, process, (6) 168; see also *Glazes*.
green, for ceramic glazes, data and curves, (6) 168–71.
hue and saturation, color diagram for reflectometer data, (6) 166.
pink and maroon, compositions and effect of firing atmosphere, (6) 174–75.
yellow, compositions and reflectance measurements in various firing atmospheres, curves and table, (6) 172–73.
**Container glass**. See *Glass bottles; Glass container*.
**Cooke triplet lens**, diagram, (6) 192.
**Cooking ware**, porcelain enameled utensils, tentative standard solubility tests of E. U. M. C., diagrams of solubility apparatus, punch and die, position of test units on hot plate, and electrical wiring equipment, (4) 114–15.
**Cooking ware**, porcelain enameled (*continued*)
**Copperheads** in enamels. See *Enamels*.
**Cordierite body**, glaze-fit, composition, (8) 233.
**Cordiness** in glass, refined method of production control, (12) 373.
**Cords and stones** in glass. See *Glass, cords, cords and stones; Stones*.
**Corning Glass Works**, optical glass types and characteristics, (6) 190.
**Cristobalite**, expansion measurements, heating and cooling cycles, (10) 289; hysteresis effect of inversion and inversion temperature and volume change, (10) 289–90; expansion test samples, properties, and inversion data, (10) 290–91.
and tridymite as source of stones in bottle glass, (11) 343; cristobalite and devitrite as source, (11) 346–47.
volume expansion in low-to-high inversion change, (10) 285.
**Crystals**, topochemically disordered, (10) 312.
**Cullet for glass**. See *Glass, cullet*.
**Cup-casting rate determination** for tests on casting slips, (4) 102.

**Debye and Hückel formula** for particle concentration, (4) 117.
**Decomposition**, thermal, effect on reactivity in magnesium formation, (10) 313.
**Definitions**, cords in glass, meaning to workmen, packing room, laboratory, and optical industry, (9) 260–61.
dielectric constant, equation for, (2) 34.
differential shrinkage, (11) 317.
dispersive power, (6) 189.
electrical terms, (6) 180.
glossary of symbols and terms for density measurements of glass, (12) 375.
hue, (6) 165.
judd, (6) 166.
lightness, (6) 165.
photoelastic constant, (3) 87.
power factor and Q factor for dielectric body, (2) 34.
refractive index, (6) 189.
saturation, (6) 165.
specific shape factor, (11) 317.
steatite, derivation and formula, (2) 33.
stress-optical coefficient, (3) 87.
thixotropy, (5) 154.
units and symbols for study of flow of suspensions, (5) 150.
**Density of glass**. See *Glass, density*.
**Devitrification** of alkali-lead oxide-dolomite lime-silica glasses, (3) 81.
**Devitrification stones** in glass. See *Stones*.
**Devitrite and cristobalite** as source of stones in glass, description and photomicrographs, (11) 346–47.
**Dictionaries**, cited, applied physics, (6) 190.
chemical solubilities, (1) 23.
inorganic chemical reactions, (1) 24.
**Dielectrics**, dielectric constants and loss factor in glassy bodies, tables, (2) 37.
dielectric materials, effect of humidity on power factor, data, (6) 177.
dielectric properties of high-frequency ceramics, effect of humidity, factors affecting, (6) 175–76.
steatite, crystalline and glassy phases, (2) 33.
**Differential thermal analysis**. See *Thermal analysis*.
**Dilatometer** for tests on volume expansion of cristobalite, (10) 287.
**Dinnerware**. See also *Chinaware; Glazes; Vitreous ware*.
glazes, strontia substitutions in, (8) 234.
glost, kiln refractories for firing, (9) 282.
**Diopside**, additions in wall-tile body; effect, firing and thermal-expansion data, curves, glaze trial results, and absorption and moisture expansion of bodies fired at different P.C.E., (11) 324–26.
as source of stone in bottle glass, description and photomicrographs, (11) 345–46.
**Dispersion constants** and specific dispersion constants, diagrams and equations, (1) 15–16.

**Earths**, fuller's, Ga., thermal curves, (3) 73.
**Eastman Kodak Co.**, optical glass types and characteristics, (6) 190.
**Einstein theoretical law** for suspension of spheres, (5) 151.
**Electrical data** on steatite dielectric test bodies, (2) 37.
**Electrical porcelain**. See *Porcelain*.
**Electrolytes** in casting slips, effect on kaolin and ball-clay content, curves, (4) 107–108, (4) 110.
effect on permeability of kaolin in casting slips, curves, (4) 100.
in glaze slips, effect of $Na_2CO_3$, curves, (7) 218.
**Enameled Utensil Manufacturers' Council**. See *Cooking ware*.
**Enamels**, abrasion resistance, abrasion indices of commercial bodies, visual ratings, and effect of $SiO_2$ content, (12) 367–68.
adherence mechanics, effect of manganese dioxide on metal precipitation at ground coat-iron interface, (11) 350–51.
clays for, carbon content vs. scattering coefficient of fired enamel, curve, (9) 256; carbon content and opacity data, (9) 257.
cleaner solutions, test methods, (3) 90.
commercial, abrasion-resistance tests, (12) 367–68.
copperheads in, effect of solubility temperature coefficient, (2) 50.
cracking tendency, effect of shrinkage and film drying behavior, (1) 26.
drying behavior, effect of soluble salts, (1) 17.
enameling iron, sheet-cleaning tests, cleaner strength and available alkali tests, (3) 91.
films and film strength, cracking tendency, effect of soluble salts, (1) 25–26; film drying behavior and shrinkage characteristics, (1) 26; packing density, effect of tearing, (1) 29; firing effect, (1) 31.
fired, clays in, properties, (9) 257–58.
frits. See *Frits*.
glass coatings on steel, absence of plastic flow in, studies, (11) 352.
iron oxide solubility, batch composition, effect of composition and temperature, (2) 46; batch weights and oxides, effect of composition, table and curves, (2) 48–49.
metal-cleaning solutions. See *Cleaner solutions*.
metal precipitation in, effect of cobalt and nickel on fired ground coats, (11) 350; at ground coat-iron interface, effect of manganese dioxide, (11) 350.
mill liquors, soluble salts in, properties, (1) 17; physicochemical behavior and possible chemical reactions, (1) 21–22.
opacity, vs. carbon content, for enamel slips, (9) 257.
porcelain, abrasion resistance, (12) 366.
porcelain, solubility test for cooking ware, (4) 113.
reflectance data, effect of carbon content and opacity of clays for, (9) 257.
sheet-steel ground-coat, solubility of iron oxide in, effect of composition and temperature, (2) 46.
shrinkage and film drying behavior, effect on cracking tendency, (1) 26.
silica content, effect on abrasion resistance, (12) 367–68.
slips, application of studies on clay, water, and electrolyte systems, (1) 25.
slips, clays for, pickup data on frits, diagrams, (9) 256–57; single clays and combined, properties, (9) 259–60; see also *Slips*.
solubility of iron oxide, batch and base compositions and composition limits, (2) 46–47; effect of composition on solubility of iron and ferric oxides, (2) 49.
soluble salts in, specific types, effect on tearing, (1) 30; see also *Soluble salts*.
tearing, visual observations during firing, (1) 25; packing density of dried enamel film, effect, (1) 29; effect of specific salts, (1) 30; relation of variables in, chart, (1) 31.
**Equilibrium diagrams**, liquidus isotherms for CaO·MgO glasses, (4) 84.
phase diagrams of CaO·MgO glasses with 12% and 16% $Na_2O$, (3) 83.
**Equilibrium systems**, bibliographies cited, Hall and Insley and Landolt, Börnstein, and Roth, (1) 24.
binary, of soluble salts, primary and derived, (1) 22–24.
possible chemical reactions between soluble salts in enamel mill liquors, (1) 22.
salt–water, diagrams, (1) 20–21.
**Expansion**, apparatus for tests, expansion bulb, thermocouple, furnace, and dilatometer, (10) 287–88.
cristobalite test samples, properties, (10) 290; inversion data on samples, (10) 291.
moisture, of bisque and glazed semivitreous bodies, curves, (12) 356–57.

**Expansion** (*continued*)
moisture, of wall-tile bodies with diopside additions, (11) 326.
silica inversions, sluggish and low-to-high types, (10) 285.
thermal, in soda-lime-silica glass, factors for calculation, (9) 262.
volume, of cristobalite in low-to-high inversion, (10) 285.

**Feldspar,** coarse-grained, as source of stones in glass, (11) 339.
as flint substitute in casting slips, (4) 106–107.
and nepheline syenite, particle-size data, (1) 5.
particle-size variations, effect on electrical porcelain body, (1) 1; data and curves for apparent distribution, (1) 6.
and talc, diopside substitutions for, in wall-tile body, (11) 324.
**Ferric oxide-$Cr_2O_3$,** X-ray studies, (6) 173.
**Ferric oxide solubility in enamel glasses.** See *Enamels, iron oxide solubility.*
**Fick's laws of diffusion,** (7) 197.
**Film strength of enamels.** See *Enamels*
**Fire-control instruments, glass for, aplanatic properties,** (6) 192.
**Fire polish of sheet glass,** nature of and measurements, (5) 145.
**Flint,** commercial, particle-size data, variations in, effect on electrical porcelain body, (1) 1; different grades, logarithmic-probability plot, data, curves, (1) 5.
feldspar as flint substitute in casting slips, (4) 106–107.
**Flint glass.** See *Glass.*
**Florida kaolins.** See *Kaolins.*
**Flow of glaze slips,** flow-stress curve, (7) 203.
of suspensions, units and symbols for study, (5) 150; properties in anisotropic particles, (5) 151; mechanism, effect of variations of concentration, particle size, rate of shear, and surface activity, (5) 156.
**Fluor-crown glass,** optical properties of, effect of variations in composition, (4) 121; tabular data, (4) 124–25.
**Fluorine in glass,** effect of $B_2O_3$ on properties, (4) 126.
in glass as $SiO_2$ substitute, effect and curves, (12) 369–70; chemical analysis and measured properties, (12) 370.
**Forsterite,** synthesis, table and reaction test, (7) 200–201.
**Freezing process** for annealing glass. See *Glass, annealing.*
**Fresnal equations** for reflecting power of dielectric, (5) 145.
**Frits, enamel,** clays for, pickup data, diagrams, (9) 256; pickup values vs. clay blends, curves, (9) 258–59.
ferric oxide in, effect on refractive index, curves, and effect of temperature on solubility, (2) 47.
for low-temperature vitreous ware, batch formulas, (12) 360.
**Fulcher equation** for measuring viscosity of glass, and constants for Fulcher equation, (5) 135–36.
**Fuller's earth.** See *Earths.*
**Furnaces.** See also *Kilns.*
atmosphere control for firing ceramic colored glazes, diagrams, (6) 166–67.
for differential thermal analyses of clays and shales, photo and wiring diagram, (3) 66.
electric, for fusion tests on chrome ore, (11) 327.
electric resistance, for atmosphere control in firing colored ceramic glazes, (6) 167.
for expansion tests on cristobalite, dilatometer, photo, (10) 287; furnace diagram and expansion bulb, (10) 288.
glass, assembly diagram for melting-rate tests, (8) 225.
glass, effect of furnace size on density values, (12) 382.
glass, refractories failures in, as source of stones, photos, (11) 331–37.
glass, stone prevention in, (11) 334.
heating chamber for measurement of electrical properties of steatite dielectrics, diagram, (2) 36.
heating rate, apparatus for constant schedules, (6) 167.
for viscosity tests on glass, high-temperature, diagram, (5) 136.
**Fused quartz,** glazed porcelain, and plate glass, surface resistivity vs. relative humidity, curves, (6) 179.
**Fusion tests** in system MgO–FeO–$Cr_2O_3$–$Al_2O_3$, (11) 327.

**Georgia clays.** See *Clays; Earths.*
**Glass,** Abbe values for pure oxides, diagram, (1) 15.
alkali-lead oxide-dolomite lime-silica, devitrification studies, raw materials and oxide compositions, (3) 81; composition, liquidus temperature, and primary phases, table, (3) 82; phase diagrams and liquidus isotherms, (3) 83–84.
alumina in, semimicro-determination method for, tables, (5) 140–41.
annealing and aging, for thermometers, Adams and Williamson equation, (2) 58.
annealing, effect on density, (9) 261.
annealing, freezing method and discontinuous freezing process, (9) 271–73.
annealing, time problem, annealing schedule, (9) 266–68; time allowance, required refractive index, (9) 273–74.
aplanatic properties in lens for fire-control instruments, (6) 192.
batch for, analysis for segregation, effect of cullet, (10) 305–306.
batch, miniature batch for comparisons, effect, (10) 307–308.
boric oxide in, effect on fluorine-containing glasses, (4) 126.
borosilicate thermometer bulb, refractive index of glass vs. thermometer reading change from aging of bulb, curve, (2) 58.
bottles for alcoholic products, effect on pH after storage three years at room temperature, (2) 53; correction, (12) 401.
bottles, autoclave chemical durability tests, pH concentration vs. chemical durability, curves, (2) 54–55.
bottles, spectroscopic studies, metallic contamination in commercial glass vs. Pyrex-brand glass, (2) 53.
bottles, stones in. See *Stones.*
chemical resistance to $H_2O$ and $H_2SO_4$ with MgO replacements for CaO, (8) 222.
colored, blisters in, source and photomicrographs, (11) 348–49.
commercial lime crystal, devitrification studies, (3) 81.
component changes in, equations for calculation, (1) 10.
composition changes in, effect on refractive index, table, (1) 12.
container, viscosity studies, (5) 129; see also *Glass, viscosity.*
control-chart method for density analysis, (12) 375; utility and limitations, (12) 387.
control of production, discussion and chart, (12) 382–84.
cords, cordiness. See also *Stones.*
"cold" or "thermal," (9) 261.
composition, separation from matrix, method, (9) 264.
cord grading of ware, density values, chart, (12) 376.
floating vs. immersed cords, Jebsen-Marwedel explanation, (5) 143.
immersed cords, surface energy in, (5) 144.
mechanical strength and stresses, types, (9) 265.
in pressed and blown ware, properties and diagnosis, (9) 260.
spherical shape, as result of surface tension, (5) 143–44.
and stones in, apparatus for $BaSO_4$ and quinolate filtration and micro-oven assembly, (5) 141.
and stones in, separation by semimicro-determination method, (5) 138; typical analysis, (5) 141.
stress and density data, interpretation, (9) 265.
various meanings to workmen, packing room, laboratory, and optical industry, (9) 260–61.
"viscosity" ridges or waves, cause, (9) 261.
and workability, refined control method, (12) 373.
crystals, and pores in ceramic body, effect of cone firing and comparison of polished specimens and powder mounts, (2) 41.
cullet additions and raw materials, effect of grain size on melting rate, (8) 225.
cullet, factory-quarried, as source of stones, (11) 332; stones in flint cullet, photo, (11) 331.
cullet in, incomplete removal, effect and errors, screen tests, (10) 306–307.
density, annealing effect, data, (9) 261.
calculations, equations and volume-constant curves, (1) 13.
changes in, thermometer arrangement for studies, (2) 61.

**Glass,** density (*continued*)
vs. chemical analysis as control method, (12) 386.
distribution curves at various melting times, (2) 44.
effect of furnace size, (12) 382.
glossary of symbols and terms for measurement, (12) 375.
Huggins' factors for calculations, (9) 262; factors in density differences, (9) 263; Archimedes' principle and sink-float methods for density measurement, (9) 263.
level, tolerable limits, composition equivalents, (12) 383; modified control limits, (12) 384.
range, statistical character of data, (12) 385–86.
statistical control charts for measurement, (12) 373.
thermal history, effect, (9) 261.
thermometers for studies, (2) 61.
values, for average and 3-sigma limits on plant furnaces, table, (12) 377; charts, (12) 378–80.
variability of plants and furnaces measured by 3-day range of density, chart, (12) 379; discussion, (12) 382; table, (12) 387.
volume constants and equation for calculation, (1) 13.
dispersion constants, equations and curves, (1) 16.
flint, clay stones in, mullite growth, photomicrographs, (11) 336–37.
flint, color equivalents, with and without tint plate, (3) 87.
fire-control instruments, aplanatic properties, (6) 192.
fire-polished, surface-tension measurements, apparatus and methods, (5) 145–48.
fluor-crown, chemical durability, thermal expansion, (4) 127–28.
fluor-crown, effect of composition variations on optical properties, (4) 121.
fluor-crown, effect of fluorine, alumina, $AlF_3 \cdot 3^1/_2\ H_2O$, $KHF_2$, and boric oxide on optical properties, (4) 124–27.
fluorine substitution for $SiO_2$ in soda-lime glass, effect, curves, (12) 369–70; chemical analysis and measured properties, (12) 370.
fractional increments, initial, for oxide additions to pure silica glass, (1) 16–17.
freezing method for annealing process, (9) 271; discontinuous freezing method, (9) 273.
frequency distribution and 3-sigma control limits, chart, (12) 385.
furnace assembly diagram for melting-rate tests, effect of grain size of raw materials and cullet additions, (8) 226; see also *Furnaces.*
glass coatings on steel, absence of plastic flow in, studies, (11) 352–54.
homogeneity, measurement of rate, (2) 42.
homogeneity, polariscopic examination of glass sections, (3) 85.
inclusions in, separation method, (5) 139.
lime, silica, daily density, and 3-day density range for ware grade, chart, (12) 381–82.
liquidus temperature, effect of MgO substitution for CaO, curves, (8) 222.
magnesia substitution for CaO in soda-lime glasses, effect on physical properties, (8) 222; effect on liquidus temperature, viscosity, and resistance to distilled water, (8) 222–24.
melting rate, effect of grain size and cullet additions, (8) 225–31.
molten, surface tension, relation to persistence of cords, (5) 143.
Multiform process for high-silica types, (10) 300.
optical, acromatic, description, proportionality, and diagram of calculation, method, (6) 189–91.
anastigmat lenses, design, (6) 194.
"bending" lens, effect on spherical aberration and coma, diagram, (6) 192; effect of cemented surface on astigmatism of lens, (6) 192.
chromatic aberration, proportionality, (6) 190.
collective and dispersive surfaces, (6) 193.
Cooke triplet lens, diagram, (6) 192.
Goerz "Dagor" lens, diagram, (6) 193.
Kodak "Ektar" f/1.5 and Kodak Telephoto Ektar f/3.8, diagrams, (6) 194.
lateral color correction, (6) 190; balancing in lens system, diagram, (6) 191.

**Glass,** optical (*continued*)
lens, chromatic aberration, calculation of separate elements in, diagram, (6) 191.
lens designer's requirements, (6) 189.
Petzval condition, formula, (6) 193; reducing Petzval sum of photographic objectives, diagrams, (6) 194.
refractive index and dispersive power, definitions, (6) 189; chart, refractive index vs. dispersive power, (6) 191.
refractive index, factors determining choice, (6) 190.
types manufactured in U. S. A., (6) 190.
optical constants, density and refractive index changes, equations for calculation, (1) 10–11.
optical properties, of fluor-crown type, (4) 124–25.
optical properties of Vycor-brand type, (10) 304.
oxide content, effect on surface tension of glass, table, (6) 187.
oxide empirical formulas, oxide and atomic equivalent bases, (2) 35.
phosphorus pentoxide substitution for $SiO_2$ and CaO·MgO in soda-lime glass, effect and curves, (12) 369–70; chemical analysis and data on properties, (12) 371.
porous, adsorption of N, curve, (10) 300; shrinkage as function of time at varying temperatures, flow of water and acetone through, curves, (10) 301.
porous, properties and process, (10) 299–300.
properties, changes in, effect of composition changes, (1) 10.
Pyrex-brand, fused $SiO_2$, and Vycor-brand types, physical properties, (10) 301.
Pyrex-brand, vs. Vycor-brand, chemical durability, (10) 303.
refractive index in annealing process, (9) 274.
refractive index change, equations, effect of density and weight, (1) 12; refractive index vs. composition, changes in, table, (1) 12.
refractive index in optical glass, studies, (6) 189.
ring-section examination, measuring apparatus, photo, (3) 88.
sheet, difference in light transmissions through two half fields plotted against analyzer angles, (5) 147.
sheet, light-transmission curves for samples of new and old glass and new glass wet-dried in air blast, (5) 147.
silica in, semimicro-determination method for, (5) 139; silica filtration assembly and tabular data, (5) 141.
sink-float methods for density measurements, (9) 263.
soda-dolomite lime-silica, fluorine and phosphorus pentoxide substitutions in, effect, (12) 369.
soda in, estimation by rapid method, (5) 148.
soda-lime and bottle, stress-optical coefficient, (3) 87.
soda-lime, grain size of raw materials and cullet additions, effect on melting rate, (8) 225–31.
soda-lime, magnesia substitution for CaO, effect on physical properties, (8) 222; effect on liquidus temperature, viscosity, and resistance to distilled water, curves, (8) 222–24.
soda-lime, strontia substitution for CaO, fused viscosity and thermal-expansion curves, (8) 237.
soda-lime-silica, homogenizing rate, centrifuge density-spread method, (2) 42.
soda-lime-silica, surface tensions, calculated, correction for, curve, (6) 187.
soda-lime-silica, thermal expansion, factors for calculations, (9) 262.
soda-silica bottle type, stones in. See *Stones.*
sodium and fluorine substitutions in, chemical analyses and viscosity data, (12) 372.
sodium silicate, change of refractive index with composition, (1) 12.
stones in. See *Glass, cords and stones; Stones.*
stress vs. birefringence, as photoelastic constant or stress-optical coefficient, (3) 87.
stress, permanent, vs. ware quality standards, (3) 88.
stress in, ring-section examination, Babinet compensator for, (3) 85.
stress in, working formulas, (3) 87.
surface tension, calculation, methods, observed-calculated values, (6) 186–88.
surface-tension measurements, spectral intensity curve of light source and filters, (5) 145; test apparatus, (5) 146.
surface tension, oxide content, effect at 1200° and 1400°C., (6) 187.
thermal history, effect of physical properties, (9) 261.
thermometers. See *Thermometers.*
transformation region, lower limit, (9) 269–70.
viscosity, composition data, maximum and minimum curves, (5) 135–36.
constants for Fulcher equation and viscosity temperature results calculated by least squares method, (5) 136.
measured vs. least squares calculated softening points, (5) 133.
measurements by high-temperature viscometer, (5) 133; by low-temperature viscometer, (5) 135; comparison of results, (5) 135; table of results, (5) 137.
temperature vs. log $\eta$ data, (5) 137.
-temperature relations for MgO replacements in lime base, (8) 222.
Vycor-brand, properties, (10) 299; physical properties, (10) 301–302; devitrification, chemical durability, operating temperatures, and lampworking properties, (10) 302–303; optical properties, infrared-transmission curves, (10) 304.
ware-quality standards vs. permanent stress, (3) 88.
workability, refined method for control during production, (12) 373.
**Glass Container Association,** Standard Testing Procedure Committee for Ring-Section Technique, tentative standard for examining and grading glass-container ring sections, (3) 85.
**Glasshouse stones.** See *Stones.*
**Glassy phase** in ceramic bodies, quantitative determination, method, (2) 40.
polished sections vs. powder mounts, results compared, (2) 41.
**Glazes.** See also *Slips.*
aging time and electrolyte additions, curves, (7) 218.
atmosphere control apparatus, diagram, and electrolytic arrangement for constant heating schedule, (6) 166–67.
autoclave test results, (12) 364.
batch formulas for frits in plant tests, (12) 363.
bisque and glazed semivitreous ware, moisture-expansion studies, curves, (12) 355–57.
Bristol, raw lead, and fritted lead borosilicate, chemical composition, test results, curves, data, (7) 211–13.
brown, composition, reflectance measurements and colors fired in various atmospheres, chart and table, (6) 173–74.
clays for, base-exchange capacity vs. coherence value, curve, (7) 219.
coherence-receptivity test method, practical uses, (7) 214.
coherence values and body receptivity, apparatus, photo, (7) 210.
colors in, effect of firing temperature, (6) 165.
consistency, control chart, (7) 216.
for cordierite body, composition and data on glaze fit, (8) 233.
deposition limit and deposition constant, (7) 219–20.
dinnerware, effect of strontia substitutions, (8) 234.
enamels, stains, effect of particle size, (1) 2.
Florida kaolin in, effect of aging and electrolyte addition on consistency, curves, (7) 218; Fla. kaolin and flint, effect on consistency and milling of slip, curves, (7) 218.
flux-block tests of cones 02 to 01 glazes fired at cone 8, photo, (12) 364.
glaze-fit problems, (8) 231–33.
glaze-slip control, detailed studies, (7) 202–203; see also *Slips.*
green, light green, firing atmospheres, chart, (6) 168; reflectance measurements, hue change vs. firing atmospheres, yellowness and lightness data, curves, (6) 169–71; physicochemical nature of changes, (6) 171.
heating schedule, electrolytic arrangement and principle, (6) 167.
impact and chipping test results, (12) 365.
improved technique and apparatus, (7) 209.
leadless and low-lead, molecular formulas and physical properties, (8) 237.
low-temperature, modulus of rupture and glazed-ring tests, data, (12) 364.
**Glazes** (*continued*)
for low-temperature vitreous ware (cone 01), physical properties, tests, (12) 359.
luster, one-fire, molecular formulas, (2) 63.
luster, "superficial" and hard, (2) 62; acid tests, (2) 64.
milling treatment, curves, (7) 217–18.
for nepheline syenite bodies, body compositions, (12) 359.
oxide composition in cones 01 and 5 glazes, (12) 364.
pink and maroon, compositions and effect of firing atmospheres, (6) 174–75.
plant trials on cone 01 low-temperature glazes, molecular and batch formulas, (12) 363.
pottery, one-fired luster, molecular formulas, (2) 63.
processing and stress developments for low-expansion bodies and durable low-temperature glazes, (8) 231–32.
raw materials, classification of variables, chart of relationships, (7) 215.
receptivity pickup for varying compositions, curves, (7) 220.
standard, and Koenig glaze, formulas, (12) 360; molecular formulas and glaze properties, (12) 361–62.
stresses in, relation to glaze fit, (8) 231; degree of stress, effect of variables, (8) 231–32.
stress variations in initial cooling, curve, (8) 233.
strontia in, molecular formulas and physical properties, tables, (8) 237.
strontia substitutions for PbO, CaO, and ZnO, cone deformation temperatures, (8) 234; fused viscosities, table and photos, (8) 235; thermal-expansion curves, (8) 236.
thermal-shock test results on plant trials, (12) 365.
thickness of application, correlation with air permeability, (7) 207; mechanical dipper for tests, diagram, (7) 208.
triaxial diagrams, glaze properties and molecular formulas for high PbO, MgO, and SrO, and high KNaO, PbO, and BaO-MgO, (12) 360–62.
for vermiculite-containing structural clayware, formula and effect, (2) 51.
wall-tile, glaze and frit formulas, (11) 324; diopside substitutions for feldspar and talc, (11) 326.
water receptivity, coherence values, and thickness, relation, (7) 207–208.
for whiteware body, glaze fit, compositions and data on, (8) 232–33.
yellow, compositions and reflectance measurements in various firing atmospheres, curves and table, (6) 172–73.
**Glost kiln refractories,** pin chipping, prevention, structure vs. performance, (9) 282–83.
**Goerz "Dagor" lens,** diagram, (6) 193.
**Grain size.** See *Particle size.*

**High-frequency ceramics,** dielectric properties, effect of humidity, (6) 175–81; see also *Dielectrics.*
**Homogeneity of glass,** soda-lime-silica, centrifuge density spread technique for measurements, (2) 42.
**Huggins' factors** for calculating glass density, (9) 262.
**Humidity,** effect on dielectric properties of high-frequency ceramics, (6) 175.
**Hunter reflectometer.** See *Reflectometers.*
**Hydrogen-ion concentration** of alcoholic products stored in glass bottles three years, test data and curves, (2) 54; autoclave durability tests, reproducibility, table, (2) 54; data on products studied, (2) 56; correction, (12) 401.
**Hydrometer** for particle-size tests, photo, (1) 7.
soil, calibration curve, (1) 3.
**Hydrometer method.** See *Methods.*

**Ice point,** changes in, effect of long-time aging at constant temperature, (2) 57.
Joule's equation for readings in thermometers, (2) 57.
**Illinois clays.** See *Clays.*
**Impact value,** formula for low-temperature vitreous ware tests, (12) 359.
**Insulators,** body composition, glaze coherence, pickup, and receptivity test data, (7) 211–13.
common solid types, surface resistivities, (6) 179.

**Inversion of cristobalite**, volume changes in low-to-high inversions, (10) 285.
**Iron, enameling**, sheet-cleaning test, cleaner strength and available alkali test, (3) 91.
**Iron oxide**, reactivities in, "sulfate" oxide + lime and "oxalate" oxide + lime, (10) 313.

**Joule's equation** for drift in ice-point readings of thermometers, (2) 57.
**Judd**, unit of color difference, definition, (6) 166.

**Kaolinite**, clean monodispersed fraction, characteristics, correlation between oxygen atoms in kaolinite sheet and in ice, diagram, (3) 80.
migration time vs. particle size, and percentage of clay, curves, (4) 119.
NaOH concentration, effect of migration velocity and zeta potential, curves, (4) 118–19.
particles, effect of charge density and total charge, (4) 120.
particles, forces acting between, calculation, (3) 80.
purified, fractionated particles, cataphoretic velocity, ultramicroscopic method, (4) 116.
**Kaolinite suspensions**, average diameter and specific surface, (5) 153.
**Kaolinite-water suspensions**, flow properties, (5) 149; data from modified MacMichael viscosimeter, (5) 159–63.
viscosity vs. volume concentration of solids, relation, (5) 152.
**Kaolins.** See also *Clays*.
in casting-slip bodies, test methods, (4) 97–111; see also *Slips, casting*.
Florida, aging time and electrolyte additions, effect on glaze consistency, curves, (7) 218.
Florida, dialyzed fraction, relation of pressure to shrinkage, percentage of water, water-film thickness, and repulsive forces between particles, table, (3) 79.
**Kilns.** See also *Furnaces*.
glost kiln refractories, pin chipping, prevention, (9) 282.
rotary, calcination of topaz ore, (6) 181; diagram, (6) 184.
**Kinetics**, reaction rates, equation, (10) 310.
**Kodak Ektar lenses**, diagrams, (6) 194.
**Kozeny permeability equation**, rate of flow, (11) 319.
**Kyanite**, kyanite brick. See also *Refractories; Topaz*.
brick with raw topaz, bonding-action temperature, (9) 278.
and kyanite-topaz bodies, volume expansion and porosity, curves, (9) 276.
raw and calcined, raw topaz as high-temperature bond, (9) 275.
-raw topaz bodies, porosity, volume change, and specific gravity, curves, (9) 277.
-topaz bodies, bonding-action temperature, effect on compressive strength, study and curves, (9) 278.
-topaz brick, standard-size, load tests, preparation and curves, (9) 279–80; preheat vs. high-temperature load tests, (9) 281.
-topaz lightweight superduty refractories, (8) 243.
topaz in, raw topaz as bond, chemical analysis and body composition, (9) 275; retention of decomposition products in calcined kyanite, curve, (9) 277–78.

**Larnite** ($\alpha$-) and **shannonite** ($\beta$-), synthesis, tabular data, (7) 200; reaction tests, (7) 201.
**Laws**, Fick's law of diffusion, (7) 197.
Stokes', for particle measurement, (1) 3.
**Lens.** See *Glass, optical*.
**Lewis, Squires, and Broughton formula** for viscosity measurements, (5) 135.
**Lillie formula**, aperiodic method for viscosity calculation, (5) 134.
**Lime** vs. magnesia, separation process, (1) 9.
**Lime-chrome mixtures**, colors of, calcined in various atmospheres, (6) 168.
**Limestone**, brucitic, description and processing, (1) 8.
**Luster glazes.** See *Glazes*.

**MacMichael viscosimeter**, modified, data on various suspensions, (5) 158–63.
**Magnesia**, granules, washing and drying process, (1) 9.
**Magnesia** (*continued*)
vs. limestone, separation process, (1) 9.
in soda-lime glasses as CaO substitute, effect on physical properties, (8) 222; effect on liquidus temperature, viscosity, and resistance to distilled water, curves, (8) 222–24.
**Magnesia spinels**, reactions with alkaline earth orthosilicates in solid state, (7) 197–202.
**Magnesioferrite**, synthesis, tabular data, (7) 200; reaction tests, (7) 202.
**Magnesium chloride**–$H_2O$, equilibrium system, diagram, (1) 21.
**Magnesium chromite**, formation from magnesia and ferrous chromite, thermal-decomposition curves, (10) 313.
**Magnesium sulfate**–$H_2O$, equilibrium system, diagram, (1) 21.
**Manganese dioxide** in enamels, effect on metal precipitation at ground coat-iron interface, (11) 350.
**Metal stones** in flint bottle glass, source, detection, and photomicrographs, (11) 348–49.
**Methods**, air separator, for steatite-talc particle-size measurements, (11) 320–21; correction, (12) 401.
Andreasen pipette, for particle-size tests, (1) 2; for measurements of steatite talc, (11) 320.
Büchner funnel vs. cup-casting, for tests on clay slips, (4) 111.
Carman, for permeability, (11) 318.
Casagrande hydrometer, for particle-size measurements of steatite talc, (11) 320.
for cleaner solution tests, soap test and free alkali and soluble soap test, (3) 91.
Fresnel's reflection laws, Pfund method, and prism method for determining fire polish on sheet glass, (5) 145.
Fulcher equation for measuring viscosity of glass, (4) 135.
glass, for cordiness and workability control in production, (12) 373.
prism, for determination of fire polish on sheet glass, (5) 145.
semimicro-determination of silica and alumina, (5) 139–40.
sink-float, for density measurements, (9) 263.
soda in, rapid estimation, (5) 148.
tentative standard, of examining and grading glass-container ring sections, (3) 85.
Harman, for clay-slip permeability, (4) 99.
hydrometer, A.S.T.M. standard method for mechanical analysis of soils cited, (1) 2.
hydrometer, for particle size of talc, (11) 318.
hydrometer vs. pipette, comparison, logarithmic-probability plot, (1) 4.
Kozeny equation of permeability, (11) 319.
Lewis, Squires, and Broughton formula for measurement of viscosity, (5) 135.
Lillie formula, aperiodic method for viscosity calculation, (5) 134.
semimicro-determination of stones and cords in glass, (5) 138.
sieve, vs. sedimentation, for particle-size tests on nonplastics, (1) 3.
soap test and free alkali soluble soap test for cleaner solutions, (3) 91.
temperature gradient, for liquidus temperatures, studies on $Na_2O$–$CaO$–$MgO$–$PbO$–$SiO_2$ glasses, (3) 81.
Turnbull and Ghering, for measurement of centrifuge density spread in soda-lime-silica glass, (2) 42.
ultramicroscopic, for cataphoretic velocity of purified, fractionated kaolin, (4) 116.
viscosity, least squares calculations of softening points, (5) 133.
**Microscopes**, polarizing, and quartz wedge for examination of glass-container ring sections, (3) 88–89.
**Mill liquors**, enamel, soluble salts in, basic physical characteristics, (1) 18–19; pyrochemical behavior, (1) 21–22; effect on film strength and tearing tendency, (1) 25.
**Minerals**, alkaline earth orthosilicates, optical properties, (7) 199.
clay, and nonclay. See *Clays, clay minerals*.
mineral compounds, synthesis, data, (7) 200.
spinels, simple synthetic types, properties, (7) 199.
**Missouri clays.** See *Clays*.
**Moisture expansion** of bisque and glazed semivitreous bodies, curves, (12) 355–57.
of wall-tile bodies with diopside additions fired at different P. C. E., (11) 326.
**Müller equation** for settling velocity of particles, (3) 77.
**Multiform process** for high-silica glass. See *Glass*.

**Nepheline syenite**, particle-size data, (1) 5.
**Nepheline syenite bodies**, compositions, cone 01 glazes for, (12) 359.
**Nepheline stones** in glass, source, analysis, photo, (11) 334–35.
**Nepheline worm** in flint glass, photo and data, (11) 335.
**New York diopside** for wall tile, (11) 324.
**Nickel oxide** with $Co_3O_4$ and $MnO_2$ in enamels, effect on metal precipitation of ground coat-iron interface, (11) 350–51.

**Ohio clays.** See *Clays*.
**One-fire luster glazes.** See *Glazes*.
**Optical constants**, density and refractive index, changes in, equations for calculation, (1) 10.
**Optical glass.** See *Glass, optical*.
**Optical properties** of fluor-crown glass, effect of variations in composition, (4) 121, (4) 124–25.
of orthosilicates, (7) 199.
of Vycor-brand glass, (10) 304.
**Orthosilicates**, alkaline earth, reactions of magnesia spinels, (7) 197; synthesis of orthosilicates and spinels, (7) 198.
**Oxide systems**, diagrams, (1) 20–21, (1) 23–24.
**Oxides**, refractory, suboxide forms, conversion at high temperatures, (10) 293.

**Particle size (grain size)**, Andreasen pipette method, instructions for use, (1) 2; for measurements of steatite talc, (11) 320.
of flint, commercial, (1) 4; of different grades of flint, feldspars, and nepheline syenite, (1) 5.
of flint, effect on ball clay in slips, (4) 108; see also *Slips, casting*.
of flint and feldspar, effect on electrical porcelain, (1) 1; of flint, feldspar, and quartz fractions, previous studies, (1) 1–2.
hydrometer method for measurement, A.S.T.M. procedure, (1) 2.
hydrometer vs. pipette methods, logarithmic-probability plot, (1) 4.
particle shape and differential shrinkage of steatite-talc bodies, (11) 317.
of raw materials and cullet additions to soda-lime glass, effect on melting rate, (8) 225.
of steatite-talc, methods for determination, (11) 320; correction, (12) 401.
suspending media, effect on settling rates, (1) 5–6.
**Particles**, cataphoretic velocity of purified and fractionated kaolin, ultramicroscopic method, (4) 116.
**Periodic tables** for systematic classification of dielectric properties, (2) 38.
**Permeability**, Carman and Kozeny methods, (11) 318–19.
of slip-casting clays, apparatus for tests, (4) 99–100.
**Petzval condition** in optical lenses, formula, (6) 193; means for reducing Petzval sum of photographic objectives, (6) 194.
**Pfund method** for determining apparent Brewsterian angle vs. angle calculated from index of refraction of glass, (5) 145.
**Phase diagrams.** See *Equilibrium diagrams; Equilibrium systems*.
**Phosphorus pentoxide** as substitute for $SiO_2$ and $CaO$·$MgO$ in soda-lime glass, effect and curves, (12) 369–70; chemical analysis and measured properties, (12) 371.
**Picrochromite**, synthesis, tabular data, reaction test, (7) 200–201.
**Pin chipping.** See *Saggers*.
**Pipette method** for particle size. See *Methods, Andreasen pipette*.
**Pittsburgh Plate Glass Co.**, optical glass types and characteristics, (6) 190.
**Plastic clays**, water film in, nature of, (3) 77.
**Plastic flow** in glass coatings on steel, tests on absence of flow, (11) 352.
**Polarizing microscope** for examination of glass-container ring sections, photo and method, (3) 88–89.
**Porcelain**, body composition, glaze coherence, and receptivity test data, (7) 211–13.
electrical, feldspar in, injurious effects on dielectric properties cited, (2) 33.

**Porcelain** (*continued*)
electrical, flint and feldspar in, effect of particle-size variations, (1) 1.
high-frequency, effect of humidity on dielectric properties, (6) 175.
vs. steatite, dissipation factor vs. temperature, curves, (6) 176.
**Porcelain Enamel Institute,** acid-resistance test, method, (12) 367.
**Porous glass.** See *Glass, porous.*
**Pottery,** glazed, one-fire luster, practical production and decorative techniques, (2) 62–64.
**Prism method** for determining fire polish on sheet glass, (5) 145.
**Pyrex-brand glass.** See *Glass, Pyrex-brand.*
**Pyrometric cone equivalent** of silica fire clay, effect of clay quality and fineness of grind, (8) 239–40.
**Pyrophyllite-talc bodies,** moisture-expansion data at various exposure conditions, tables, (12) 356.

**Quartz,** grain size, effect on body contraction and expansion, previous work cited, (1) 1–2.
preheating of, effect on reactivity, preheating in $O_2$, $SO_3$, air, and $SO_2$, curves, (10) 313.
as source of stones in bottle glass, (11) 342.

**Rankin and Merwin diagram,** $MgO–Al_2O_3$, cited, (11) 327; curve, (11) 329.
**Reactions,** $Ag_2HgI_4$, mechanism of formation, diagram, (10) 311.
chemical, bibliography, (1) 23–24.
chemical potential of diffusing particles during, (10) 309.
kinetic treatment for reaction rate, (10) 310.
rates, topochemical factors in, influence, (10) 311–12.
reaction constants, diagram and equation, (1) 14–15.
reaction mixtures, increased adsorption, curves, (10) 315.
reaction products, fresh, expanded lattice data, (10) 311.
simple additive, course of, period of primary reaction, (10) 314.
between solid salts, topochemical factors, (10) 308.
in solid state, rate-determining factors, (10) 308.
**Receptivity data** for slip glazes. See *Glazes: Slips.*
**Reflectance,** measurements of green, yellow, and brown glazes, effect of various firing atmospheres, (6) 170, (6) 172, (6) 174.
**Reflectometer,** Hunter, for color measurement of glazed surfaces, use, (6) 168.
**Refraction,** refraction constants and specific refraction constants, diagrams and equations, (1) 14–15.
**Refractive index** of glass, calculation of changes in, equations, (1) 11; vs. composition changes, table, (1) 12.
**Refractories,** brucite magnesia for, source, processing, and uses, (1) 8–10.
clays for. See *Clays.*
fusion-cast, with high-chrome ore content, tests, (11) 327.
fusion experiments in system $MgO–FeO–Cr_2O_3–Al_2O_3$, (11) 327.
glost kiln, pin chipping, cause and prevention, linear expansion curves, (9) 283–84.
high-grade mullite-type, topaz ore as substitute for India kyanite, (6) 181.
Illinois plants, thermal analyses of fire clays, curves, (3) 74–75.
kyanite-topaz brick, load-test results, (9) 281; see also *Kyanite.*
kyanite-topaz, lightweight compositions, effect of plastic kaolin additions, properties before and after reheat tests, (8) 242; see also *Refractories, lightweight.*
kyanite-topaz, properties after preheat at 1500°C., variable temperature load-test results, (9) 280.
lightweight, superduty, Bur. Mines vs. commercial test samples, reheat test properties at 1600°C., (8) 244; load tests, constant and variable, tables, (8) 245.
lightweight, superduty, porosity and density curves, (8) 243; composition and reheat test results, tables, (8) 244.
load tests for kyanite-topaz brick, (9) 279–81.
pyrometric cone equivalent (P.C.E.), effect of clay content and quality, (8) 240.
in Owens revolving pot, drips and stalactites, photo, (11) 331.
**Refractories** (*continued*)
refractory origin of stones in glass, (11) 334.
refractory oxides, suboxide forms, conversion at high temperatures, (10) 293; significance in study of refractories and slags, (10) 297.
silica brick, mineral analysis and volume expansion of cristobalite, (10) 285.
silica fire clay, chemical analysis of raw materials, effect of fineness of grind on P.C.E., tables, (8) 239–40.
and slags, suboxide forms, significance, (10) 297.
topaz, domestic kyanite, and synthetic mullite corundum as substitution for India kyanite, raw topaz as high-temperature bond for domestic kyanite, IV, (9) 275.
topaz ore, calcination in rotary kiln, (6) 181; see also *Topaz.*
zirconium, in glass furnaces as source of stones, (11) 338.
**Refractory stones** in glass. See *Stones.*
**Ring sections** of glass containers, tentative standard procedure for examination, (3) 86.
**Rotary kilns.** See *Kilns.*
**Rutile bodies,** compacting pressure vs. dimensions, dielectric constant, and dissipation factor, tables, (6) 178.
dielectric constant and temperature dependence, curve, (6) 179.
dissipation factor and dielectric constant vs. temperature, (6) 176; variation of dissipation factor and capacitance with temperature, curves, (6) 176.

**Saggers,** pin chipping, prevention, (9) 282; structure vs. performance, relation, (9) 283; refractory bases, expansion curves, (9) 283–84.
**Sanitary ware,** body compositions, glaze coherence, pickup, and receptivity test data, (7) 211–13.
**Sedimentation,** analyses, effect of non-polar liquids, (1) 7.
methods for particle-size measurements of steatite talcs, (11) 321; correction, (12) 401.
**Semivitreous ware.** See *Vitreous ware, semivitreous.*
**Shales** from northern and southern Ill., thermal curves, (3) 74–75; see also *Clays; Kaolins.*
**Shrinkage,** differential, and particle shape of steatite talc bodies, (11) 317.
**Sieve method.** See *Methods.*
**Silica** and alumina in glass, semimicro-determination methods, (5) 139–40.
conversion to silicon monoxide at high temperatures, SiO history, (10) 293–94; properties, uses, and experiments on SiO, (10) 294–95.
in enamels, effect on abrasion resistance, (12) 367–68.
expansion data, properties of test samples, inversion data, (10) 290–91.
inversions, sluggish and low-to-high types, (10) 285.
suboxide forms, CrO, properties and functions, (10) 296.
**Silica brick.** See *Refractories.*
**Silica fire clay,** pyrometric cone equivalent, effect of clay fineness of grind and quality, (8) 239–40.
**Silicon monoxide,** vitreous, decomposition products, photomicrograph, (10) 294; powdery sublimate of SiO, photo, (10) 294.
**Sink-float methods** for glass density measurements, (9) 263.
**Slips,** casting, aging time for, effect on kaolin and ball clays, curves, (4) 105.
casting, amount and temperature, effect on kaolin, curves and data, (4) 104.
casting-rate determination, cup method, (4) 112.
clay-flint ratio, effect on kaolin and feldspar substitution for flint, (4) 107.
control methods, (4) 103.
mixing time, mixing sequence, and variation of vacuum pressure, effect, curves and table, (4) 106.
particle numbers and spacing in suspension, calculation, (5) 157.
plaster of Paris mold for, design, (4) 102; mold for cup-casting rate determination, (4) 102.
viscosity curves, actual vs. calculated, viscosity values, (5) 156–57; viscosity and yield-point calculations, (5) 157.
water content, effect, curves, (4) 103.
clay, cup method for casting rate, (4) 112.
**Slips** (*continued*)
clays for, casting characteristics, improved methods, (4) 97.
coherence test apparatus, diagram, process, (7) 204.
drier and stirrer for preparation, (4) 99.
electrolytes in, effect on kaolin and ball clay content, curves, (4) 107–108, (4) 110.
enamel, clays for, physical and chemical properties, X-ray patterns, (9) 253–254.
enamel, clays in, single clays and in combination, properties, (9) 258–60.
flint in, effect of particle size on ball clays, curves and data, (4) 108.
glaze, coherence, surface tension, thickness, and firing, test results, (7) 204–207; see also *Glazes.*
glaze, specifications, flow-stress curve, (7) 203.
properties, Büchner funnel vs. cup-casting tests, thixotropy and shrinkage data, (4) 109; properties of bodies and tests compared, (4) 111.
surface-tension apparatus, and tests, (7) 204–205.
thixotropy tests, Stormer viscosimeter for, (4) 99; test curves on casting slips, (4) 109.
viscosity, apparent, at different rates of shear, curves, (5) 152.
**Smoluchowski formula** for particle concentration, (4) 117.
**Soda** in glasses, rapid method for estimating, (5) 148.
**Soda-dolomite lime-silica glasses,** fluorine and phosphorus pentoxide substitutions in, effect, (12) 369.
**Sodium borate**–$H_2O$, equilibrium system, diagrams, (1) 20.
**Sodium carbonate**–$H_2O$, equilibrium system, diagram, (1) 20; $NaCO_3$–NaF, diagram, (1) 22; $NaCO_3$–NaOH, diagram, (1) 23.
**Sodium fluoride**–$H_2O$, equilibrium system, diagram, (1) 20; NaF–$NaSO_4$, diagram, (1) 22.
**Sodium hydroxide** in cleaner solutions, formulas for calculation, (3) 93.
–$H_2O$, equilibrium system, diagram, (1) 21.
**Sodium nitrite**–$H_2O$, equilibrium system, diagram, (1) 21.
**Sodium pyrophosphate**–$H_2O$, equilibrium system, diagram, (1) 20.
**Sodium sulfate**–$H_2O$, equilibrium system, diagram, (1) 21; $Na_2SO_4$–$MgSO_4$ and $Na_2SO_4$–$Na_2CO_3$, diagrams, (1) 23.
**Soil hydrometer,** calibration curve, (1) 3.
**Solids** in topochemical states, source, (10) 312.
**Solid state,** alkaline earth orthosilicates, reactions of simple magnesia spinels with, (7) 197–202.
reaction rates between solid salts, effect of topochemical factors, (10) 308.
**Solubility test** for enameled cooking ware. See *Cooking ware.*
**Soluble salts** in enamel mill liquors, effect on enamel film strength and tearing tendency, (1) 25; specific types, effect on tearing of enamels, (1) 30.
in enamel mill liquors, fundamental properties, (1) 17; basic physical characteristics, tables, (1) 18–19; pyrochemical behavior, (1) 21; possible chemical reactions, formulas, (1) 22.
ionization behavior, reactions between, effect on clay–water systems, (1) 20.
**Sound-absorption,** tests on plastic-process tile with vermiculite, (2) 51.
**Specific shape factor,** calculation, (11) 317–18.
**Specific surface,** equation for calculation, (1) 4.
**Spinel,** formation, schematic mechanism, (10) 314.
and orthosilicates, synthesis of, (7) 198.
synthesis, tables and reaction tests, (7) 200–201.
synthetic, properties, (7) 199.
**Steatite, Bakelite, and mica,** variation of dissipation factor with frequency, curves, (6) 177.
definition and formula, (2) 33.
dissipation factor vs. maximum firing temperature, curve, (6) 179.
vs. porcelain, dissipation factor vs. temperature, curves, (6) 176.
and rutile materials, variation of dissipation factor with frequency, curves, (6) 177.
surface resistivity vs. air pressure for varying relative humidity, (6) 180.
**Steatite bodies** with talc and various powdered glasses, (2) 34; empirical formulas of glasses, (2) 35.

**Steatite dielectrics,** absorption measurements and electrical tests, (2) 35–36; furnace for measurements, diagram, (2) 36.
crystalline and glassy phases, (2) 33.
electrical properties, definitions and formulas, (2) 34; tabular data, (2) 37.
periodic table for systematic classification, (2) 38.
**Steatite-talc,** air-separator method for particle-size measurements, disadvantages, (11) 320–21; correction, (12) 401.
bodies, particle shape and differential shrinkage, correlation, (11) 317.
particle-size distribution, (11) 320.
**Steel,** glass coatings on, absence of plastic flow in, studies, (11) 352–54.
**Stokes' law** for particle measurement, (1) 3.
**Stones.** See also *Glass, cords and stones.*
batch, source, prevention, photomicrographs, (11) 338–40.
in bottle glass, quartz as source, (11) 342.
clay, chemical analysis, source, and photos, (11) 335–36.
cullet as source, flint samples, photo and discussion, (11) 331, (11) 333.
devitrification types, identification, (11) 330; in bottle glass, source, cause, photomicrographs, (11) 340–42.
devitrite and cristobalite as source in bottle glass, description and photomicrographs, (11) 346–47.
diopside as source in bottle glass, (11) 345–46.
in flint bottle glass, (11) 347–49.
in flint cullet, source, photo, (11) 331.
in flint glass, nepheline worm, photos and data, (11) 335.
from furnace refractories, origin and source, (11) 333–34.
in glass, bibliography and footnote citations, (11) 330–50.
in glass, opaque vs. translucent, differentiation, (11) 330.
glasshouse, in flint, amber and green bottle glass, source, identification, and elimination, (11) 330–50.
glasshouse, in melting soda-lime bottle glass, types, description, and source, photomicrographs, (11) 330.
metal types in flint bottle glass, detection and photomicrographs, (11) 348–49.
in molten cast refractory, description and photomicrograph, (11) 336–37.
nepheline type, photos, sources, and chemical analysis, (11) 334–35.
silica refractory, chemical analysis, source, photomicrographs, (11) 338.
special refractory, effect of zirconium, (11) 338.
sulfate in flint bottle glass, source and photomicrographs, (11) 347–48.
tridymite and cristobalite as source in bottle glass, (11) 343.
wollastonite as source in bottle glass, description and photomicrographs, (11) 344–45.
**Stormer viscosimeter** for permeability tests on casting clays, (4) 99–100; thixotropy test curves, (4) 109.
**Stress** in glass and glazes. See *Glass; Glazes.*
**Strontia** in dinnerware glazes as PbO, CaO, and ZnO replacement, (8) 234.
**Strontium,** calcium, and barium compounds, properties and eutectic melting temperatures, (8) 234.
**Strontium orthosilicate,** synthesis, tabular data, reaction tests, (7) 200–201.
**Structural materials,** vermiculite as raw material for, (2) 50.
**Suboxide forms** of refractory oxides. See *Refractories, refractory oxides.*
**Sulfate stones** in flint bottle glass, source and photomicrographs, (11) 347–48.
**Surface tension** of glaze slips, curves for Tenn. ball clay and Texas clay, (7) 296.
**Suspensions,** kaolinite-water, flow properties, (5) 149; flow properties of suspended spheres, Einstein, Hatschek, and Arrhenius laws for, (5) 151.
particles in, at varying concentrations, (5) 156.
**Synthesis** of spinels and orthosilicates, (7) 198; of compounds, table, (7) 200.
**Systems,** clay–water, effect of various soluble salts, (1) 20.
$MgO$–$Al_2O_3$, Rankin and Merwin diagram cited, (11) 327; curve, (11) 329.
$MgO$–$FeO$–$Cr_2O_3$–$Al_2O_3$, fusion experiments in, (11) 327.

**Talc-clay body,** shrinkage curves, effect of particle size, (11) 322.
commercial, particle-size, comparisons, (11) 321.
and feldspar, diopside substitution in wall-tile body, (11) 324.
Manchurian, for steatite dielectrics, chemical analysis, (2) 35.
materials, particle type, specific surface, and volume data, (11) 319.
particle size, hyrometer method for determination, (11) 318.
-pyrophyllite bodies, moisture-expansion data at various exposure conditions, tables, (12) 356.
sedimentation methods for measurements on particle size, (11) 321.
shrinkage control, effect of particle shape and blends, (11) 317.
specific shape factor and shrinkage, statistical data, (11) 319.
standard and fine grinds, particle-size distribution curves, (11) 322; shrinkage curves, (11) 323.
**Tearing** of enamels. See *Enamels.*
**Telescope design,** (6) 191.
**Thermal analysis,** differential, application to prospecting, (3) 74–75.
differential, of clays and shales, control and prospecting method, (3) 65.
differential, of common clay-minerals and nonclay minerals in clays and shales, (3) 68; of prepared mixtures of clay and nonclay minerals, (3) 69; thermal curves for synthetic clay mixtures, (3) 70.
**Thermal decomposition,** effect on reactivity in magnesium formation, (10) 313.
**Thermal expansion.** See *Expansion.*
**Thermometers** for density changes in glass, (2) 61.
drift in ice-point readings, reversals and complex effects, (2) 60.
glass, ice point, effect of aging, (2) 57; ice-point change vs. time, curves, (2) 59.
**Thixotropy,** definition, equation for thixotropy of suspension, (5) 154; curves for thixotropy of H-kaolinite fractions, (5) 154.
method of determination, diagram, (5) 150.
tests on clays for casting slips, Stormer viscosimeter for, (4) 99; test curves, (4) 109.
**Tin-chrome system,** colors developed, (6) 174.
**Topaz,** calcination in rotary kilns, data for, (6) 182.
calcined and raw, screen analyses, physical properties, (6) 183; chemical analyses, raw materials, products, (6) 182; dust and volatilization losses, (6) 185.
in kyanite bodies. See also *Kyanite.*
**Topaz** (*continued*)
raw, as high-temperature bond for raw and calcined kyanite, (2) 275.
**Topochemical factors,** significance on rate of reactions between solid salts, (10) 308.
**Tridymite** and cristobalite as source of stones in bottle glass, (11) 343.
**Turnbull and Ghering method,** for measurement of centrifuge density spread in soda-lime-silica glass, (2) 42.

**Vermiculite, expanded,** for structural clayware, uses, and tests, (2) 50.
in structural clayware, thermal-conductivity and sound-absorption tests, (2) 51.
**Viscometers,** high-temperature, concentric cylinder, methods of use, photos, (5) 130–31.
low-temperature, method of use, photo, (5) 132–33.
MacMichael, modified, data on various suspensions, (5) 158–63.
Stormer, for thixotropy tests on clays, (4) 99; test curves, (4) 109.
**Viscosity** of container glass, (5) 129; high- and low-temperature viscometers for measurements, photos, (5) 130–32; maximum and minimum viscosity curves, diagram of high-temperature viscosity furnace, (5) 136.
high-temperature measurements, methods of calculation, (5) 133–34.
of suspension, relation to concentration of solid, formula for, (5) 152.
**Vitreous ware.** See also *Chinaware; Dinnerware.*
low-temperature, cone 01 glazes, (12) 358.
semivitreous, clay-flint-feldspar body, moisture-expansion data, (12) 357.
semivitreous, moisture-expansion data, on bisque and glazed bodies, (12) 355; observed and ultimate expansion at cone 9, curve, (12) 357.
**Volume-expansion measurements** on cristobalite. See *Expansion.*
**Vycor-brand glasses.** See *Glass, Vycor-brand.*

**Wall tile,** colors in, effect of diopside substitutions for talc and feldspar, (11) 325.
diopside additions and substitutions for talc and feldspar, compositions, firing temperatures, thermal-expansion, glaze trial results, moisture-expansion, and absorption data, (11) 324–36.
glaze and frit formulas, glaze trials, test results, (11) 324, (11) 326.
**Water film** in plastic clay, nature, determination, and stability, (3) 77–79.
**Whiteware.** See also *Chinaware; Porcelain, electrical; Vitreous ware.*
glassy bond in, quantitative determination, method, (2) 40.
glazes. See *Glazes; Slips.*
petrographic study of polished sections and powder mounts, for glassy phase, crystals, and pores, (2) 40–41.
semivitreous bodies, moisture-expansion data at varying storage periods, (12) 355–57.
**Wollastonite** as source of stones in bottle glass, photomicrographs, (11) 344–45.

**Zinc-aluminum-spinel,** formation, photo, (10) 309.
-iron-spinel formation, intermediate stages in, diagram, (10) 314.
**Zinc oxide** and $\alpha$-iron oxide, lattice imperfections, tabular data, (10) 312.

## FOOTNOTE CITATIONS INDEX TO THE JOURNAL
### Volume 27, 1944

The reference number in parentheses refers to the *monthly* issue of the *Journal;* the number following is the *page* number.

**Abramson, H. A.,** determination of electrical charge of surfaces by microscope method of electrophoresis, (4) 116.
**Adams, L. H., and Williamson, E. D.,** annealing of glass, (2) 58, (5) 146, (9) 272.
**Albers-Schönberg, E.,** Hochfrequenskeramik (high-frequency ceramic products), (6) 176.
**Albro, A. B.,** analysis of silicon compounds, (10) 293.
**American Institute of Electrical Engineers,** American standard definitions of electrical terms, (6) 180.
**American Society of Heating and Ventilating Engineers,** Guide on thermal conductivity of materials, (2) 51.
**American Society for Testing Materials,** Nonmetallic materials, III, 1942, (6) 180; standard method of mechanical analysis of soils, A.S.T.M. designation D422–39, (1) 2; Symposium on new met hod particle-size determination in subsieve range, (1) 2; tentative method of hydrostatic pressure test on glass containers, C147–40T, (3) 86; tentative method of polariscopic examination of glass containers, C148–40T, (3) 85; tentative method of thermal-shock test on containers, C149–40T, (3) 85.
**American Standards Association,** control-

chart method of controlling quality during production, (12) 374.
**Andreasen, A. H. M.**, grinding of materials, theoretical and experimental research on particle-size determinations incident to disintegration process, (1) 2; validity of Stokes' law for nonspherical particles, (1) 4.
**Andrews, A. I.**, Enamels, (1) 28, (2) 46, (3) 90, (11) 354.
**Andrews, A. I., and Cook, R. L.**, Enamel laboratory manual, (2) 48.
**Andrews, A. I., and Swift, H. R.**, iron oxide in enamel glass as dissolved from metal base, (2) 46.
**Andrews, A. I., and Zwermann, C. H.**, fundamentals of colors in porcelain enamels, (6) 173.
**Annual Tables** of Constants and Numerical Data, Vol. IV, (1) 23.
**Arrhenius, Svante**, Innere Reibung verdünnter wässeriger Lösungen, (5) 151.
**Athavale, V. T., and Jatkar, S. K. K.**, reactions of chromates at high temperatures, decomposition of calcium chromate, III, (6) 169.
**Austin, J. B.**, methods of representing distribution of particle size, (1) 4.

**Babcock, C. L.**, surface-tension measurements on molten glass by modified dipping-cylinder method, (6) 186; viscosity and electrical conductivity of molten glasses, (5) 134.
**Bacon, F. R., and Burch, O. G.**, effect of time and temperature on accelerated chemical durability tests on commercial glass bottles, (2) 53.
**Badger, A. E.**, effect of various gaseous atmospheres on vitrification of ceramic bodies, (10) 297.
**Badger, A. E., Parmelee, C. W., and Williams, A. E.**, surface tension of various molten glasses, (6) 186.
**Badger, A. E., and Pinnow, H. R.**, vanadium and chromium oxides in refractories as aids in elimination of cords in glass, (5) 143.
**Badger, A. E., Uhrmann, C. J., and Ottoson, A. C.**, use of lead oxide in glassmaking refractories, (5) 143.
**Balinkin, I. A.**, color symposium, measurement and designation of small color differences, V, (6) 166.
**Baly, E. C. C.**, Spectroscopy, (4) 121.
**Barr, Guy**, Monograph of viscometry, (5) 164.
**Barrer, R. M.**, diffusion in and through solids, (7) 197.
**Barth, T. F. W.**, cristobalite structures: high cristobalite, I, (10) 286; low cristobalite, II, (10) 286.
**Baumann, H. N., Jr.**, X-ray diffraction examination of material having composition $SiO_2$, (10) 294.
**Bauer, T. D.**, Soil physics, (1) 5.
**Bazilevich, A.**, synthesis of spinels, (7) 198.
**Beam, J. R.**, effect of opacifiers on fused viscosity of feldspathic glazes, (8) 234.
**Bell, W. C.**, evaluation of glaze-fit test methods, (8) 236.
**Bellière, Marcel**, crystallization of pseudowollastonite by devitrification of glasses, (11) 330; formation of cristobalite as octahedra in devitrified glass, (11) 350.
**Bénard, J., and Chaudron, Georges**, preparation of ferrites by substitution for ferrous irons in magnetite, (7) 198.
**Benner, R. C., Baumann, H. N., Jr., and Easter, G. J.**, refractory and abrasive material and process of making, (11) 327.
**Bennett, A. L.**, special depth gauge for measurement and control of ceramic coatings, (7) 203.
**Bennett, H. F.**, telephoto objective, (6) 194.
**Berberich, L. J., and Bell, M. E.**, dielectric properties of rutile form of $TiO_2$, (6) 176.
**Berenz, H., and Gingold, J.**, insulator, (2) 34.
**Berger, E., and Freundel, O.**, oxide compositions of fluor-crown glasses, (4) 121.
**Bernard, Jacques**, Étude de la Decomposition du Protoxide de Fer et de ses Solutions Solides, (11) 350.
**Bhatanger, S. S., Cameron, A., Harbard, E. H., Kapur, P. L., King, A., and Prakash, Brahm**, magnetic susceptibilities of metallic oxides with special reference to those of chromium and manganese, (6) 169.
**Biltz, W., and Ehrlich, P.**, volatilization of silicon as lower oxide, (10) 294.
**Blair, G. W. S.**, measurements of plasticity of clays, (7) 202.
**Blair, T.**, certain accessory products of blast furnaces, (10) 298.
**Blanc, L., and Chaudron, G.**, transformations and oxidation of sesquioxides of chromium, (6) 169.
**Bleininger, A. V., and Hornung, M. R.**, notes on casting, (4) 98.
**Bleininger, A. V., and Riddle, F. H.**, special spark-plug porcelains, (2) 33.
**Bock, F.**, several innovations in technical utilization of silica, (10) 293.
**Boericke, F. S.**, equilibria in reduction of chromic oxide by carbon and their relation to decarburization of chromium and ferrochrome, (10) 296.
**Bonhoeffer, K. F.**, existence of gaseous silicon monoxide, (10) 294.
**Benthron, K. J. A., and Durrer, Robert**, studies for determination of melting diagrams of system $Al_2O_3$–$Cr_2O_3$–MgO, (7) 199.
**Boonton Radio Corp.**, Instructions and manual of radio frequency measurements, (2) 34.
**Bouyoucos, G. J.**, comparison between pipette method and hydrometer method for mechanical analyses of soil, (1) 2; hydrometer as new and rapid method for determining colloidal content of soils, (1) 2; sensitive hydrometer for determining small amounts of clay or colloids in soils, (1) 2.
**Bowen, N. L.**, identification of stones in glass, (11) 330.
**Bowen, N. L., and Greig, J. W.**, system $Al_2O_3$–$SiO_2$, (11) 350.
**Bradley, R. S., Schroeder, F. W., and Keller, W. D.**, study of refractory properties of topaz, (6) 181, (9) 278.
**Bragg, W. L.**, Atomic structures of minerals, (10) 285.
**Brauer, G.**, oxides of columbium, (10) 297.
**Brunauer, S., Emmett, P. H., and Teller, E.**, adsorption of gases in multimolecular layers, (10) 300.
**Bryant, E. E.**, controlled boiling acid test for porcelain enamels, (4) 113.
**Büssem, W., Schusterius, Carl, and Stuckhardt, Karl**, constitution of steatite, glass phase, II, (2) 33.

**Cameron, A., Harbard, E. H., and King, A.**, nonstoichiometric oxides, oxides of chromium, I, (6) 169.
**Canfield, J. J.**, copperheading in enamel, causes and methods of prevention, (2) 46.
**Carman, P. C.**, determination of specific surface of powders, I, (11) 318; shape and surface of fine powders by permeability method, (1) 2.
**Carter, W. K., and King, R. M.**, sodium aluminate as an electrolyte for casting-slip control, (4) 98.
**Chesters, J. H.**, all-basic open hearth, II, (7) 202.
**Chesters, J. H., and Parmelee, C. W.**, measurement of reaction rates at high temperatures, (7) 198.
**Clark, G. L., Ally, Abde, and Badger, A. E.**, lattice dimensions of spinels, (7) 199.
**Clark, G. L., Howe, E. E., and Badger, A. E.**, lattice dimensions of solid solutions in the system MgO–$Al_2O_3$, (7) 199.
**Cohn, W. M.**, problem of heat economy in ceramic industry, II, (10) 286.
**Cole, S. S.**, effect of size of grain on properties of silica cement, (8) 241; variations in P.C.E. of silica cements and fire clays, (8) 241.
**Cole, S. S., Scholes, S. R., and Amberg, C. R.**, correction in specific gravity and unit cell size of $Na_2O \cdot B_2O_3$, (1) 23.
**Comey, A. M., and Hahn, D. A.**, Dictionary of chemical solubilities, (1) 23.
**Conrady, A. E.**, microscopic optics in Dictionary of applied physics, Vol. IV, (6) 190.
**Cook, H. L.**, note on solubility of enamel frit in mill water, (1) 25; observations on effect of various electrolytes added to enamel suspensions made with and without clay, (1) 25.
**Cooke, R. D.**, effect of furnace atmosphere on firing of enamel, (6) 165; study of soluble salts in enamels, (1) 25.
**Cosgrove, J. M.**, spot test as aid in rapid evaluation of cleaners, (3) 90.

**Danielson, R. R.**, effect of soluble salts on properties of enamels, (1) 25.
**Davis, H. E., and Lueders, R. L.**, simplification of ring method for determining glaze stresses, (12) 355.
**Debye, P., and Hückel, E.**, Bemerkungen zu einem Satze über Kataphoretische Wanderungsgeschwindigkeit suspentister Teilchen, (4) 117.
**Dickinson, H. C.**, heat-treatment of high-temperature mercurial thermometers, (2) 57.
**Dieckmann, T., and Haaf, O.**, notice on chromous oxide, (10) 296.
**Dietzel, A.**, methods for investigation of little stones and knobs in glass, (11) 350; practical importance and calculation of surface tension in glasses, glazes, and enamels, (6) 186.
**Dilaktorskii, N. L.**, synthesis of some minerals of the spinel group, (7) 198.
**Dodd, A. E.**, action of water vapour on silica brick at high temperatures and possible industrial significance, (10) 297.
**Drude, P.**, Theory of optics, (5) 145.

**Earhart, W. H.**, use of N. C. kaolin in casting bodies, (4) 98; use of washed N. C. kaolin as ingredient in porcelain bodies, (4) 98.
**Ebelman, M.**, cited on method of spinel synthesis, (7) 198.
**Einstein, A.**, Neue Bestimmung der Molekûldimensionen, (5) 151.
**Eirich, F., Bunzl, M., and Margaretha, H.**, Viskosität Suspensionen und Lösungen, Kugelsuspensionen (viscosity of globular suspensions), IV, (5) 151.
**Eirich, F., Margaretha, H., and Bunzl, M.**, Viskosität von Suspensionen, Viskosität von Stäbchensuspensionen (viscosity of acicular suspensions), VI, (5) 152.
**Eitel, W.**, flow currents in automatic glassworking in Owens process, special current configuration in finished bottles, (3) 86; flowing processes in automatic manufacture of bottles by feeder process with Owens Bottle Company machine, (3) 86; processes of flow in automatic working of glass in Owens process, (3) 86; studies of flow conditions in automatic glassworking by feeder-fed (Lynch process) machines, (3) 86.
**Ellis, R.**, Eigenschaften der Olemulsionen und electrische Ladung, (4) 116.
**Emmett, P. H.**, measurement of surface areas of finely divided or porous solids by low-temperature adsorption isotherms, (10) 299; new method for measuring surface areas of finely divided materials and for determining size of particles, (1) 2.
**Emmett, P. H., and DeWitt, T. W.**, low-temperature adsorption of nitrogen, oxygen, argon, hydrogen, *n*-butane and carbon dioxide, (10) 300.
**Endell, Kurd, and Rieke, R.**, transformation of silicic anhydride at high temperatures, (10) 286.
**English, S., and Turner, W. E. S.**, heat expansion of soda-lime glasses, (2) 62.
**Ewald, P. P., and Hermann, C.**, Strukturbericht, 1913–1928, (10) 286.
**Ewing, W. W.**, specific surface of pigments by adsorption from solution, (1) 2.

**Fang, S.-M.**, crystal structure of sodium metaborate, $NaBO_2$, (1) 24.
**Feild, A. L.**, method for measuring viscosity of blast-furnace slag at high temperature, (5) 130.
**Fenner, C. N.**, relations between tridymite and cristobalite, (11) 350; stability relations of silica minerals, (10) 286.
**Ferguson, W. F. C.**, chromium oxide and vanadium oxide band spectra, (10) 296.
**Ferry, J. D., and Parks, G. S.**, viscous properties of polyisobutylene, (5) 134.
**Fisk, H. G., and McCaughey, W. J.**, equilibrium studies in systems containing magnesium oxide, iron oxide, and magnesium aluminate, (7) 199.
**Flusin, R.**, silicon monoxide and oxycarbides, "Monox" and "Fibrox," (10) 293.
**Frame, W. M.**, Casing settling depths not assured by physical properties of steel, (11) 353.
**Freundlich, H., and Jones, A. D.**, sedimentation, volume, dilatency, thixotropic, and plastic properties of concentrated suspensions, (5) 151.
**Freundlich, H., and Röder, H. L.**, dilatency and its relation to thixotropy, (5) 151.
**Fricke, R., and Ackermann, P.**, heat content and lattice structure of active ferric oxide, (10) 312; thermal and X-ray characterization of irregular lattice distortions in zinc oxide, (10) 312.
**Fricke, R., and Dürr, W.**, active substances, thermochemical and X-ray investigation

of reactions during formation of zinc-iron spinel from active zinc oxide and various kinds of ferric oxide, XXXVII, (10) 314.
**Fricke, R., and Gwinner, E.,** quantitative interpretation of irregular lattice distortions and impurities of amorphous material in active substances, (10) 312.
**Fritz, E. H., and Donsenwirth, A. L.,** observations in casting of heavy electrical porcelains, (4) 97.
**Fulcher, G. S.,** analysis of recent measurements of viscosity of glasses, (5) 133.

**Geller, R. F., and Creamer, A. S.,** moisture expansion of ceramic whiteware, (12) 355; note on moisture expansion of ceramic whiteware in storage and in service, (12) 355.
**Geller, R. F., Evans, D. N., and Creamer, A. S.,** effects of particle size of a potter's flint and feldspar in whiteware, (1) 1.
**Getman, F. H.,** Outlines of theoretical chemistry, (12) 358.
**Ghering, L. G., and Knight, M. A.,** properties and diagnosis of cords in pressed and blown glassware, (12) 374.
**Ghosh, C.,** band spectrum of chromium oxide, (10) 296.
**Gifford, J. W.,** quartz-fluorite for achromatic lens, (4) 121.
**Glass Container Association of America,** polariscopic examination of glass container sections, (9) 265.
**Glazebrook, R.,** Dictionary of applied physics, Vol. IV, (6) 190.
**Goodeve, C. F., and Whitfield, G. W.,** measurement of thixotropy in absolute units, (5) 150.
**Gottfried, C., and Shossberger, F.,** Strukturbericht, Vol. III, 1933–1934, (10) 286.
**Goucher, F. S., and Ward, H.,** problem in viscosity, thickness of liquid films found on solid surfaces under dynamic conditions, (7) 203.
**Goudge, M. F.,** preliminary report on brucite deposits in Ontario and Quebec and their commercial possibilities, (1) 8.
**Granat, I. Y.,** reduction of chromium oxide, (10) 296.
**Greig, J. W.,** formation of mullite from cyanite, andalusite, and sillimanite, (6) 181.
**Greig, J. W., Posnjak, E., Merwin, H. E., and Sosman, R. B.,** equilibrium relationships of $Fe_3O_4$, $Fe_2O_3$, and oxygen, (6) 173.
**Griffin, R. C.,** Technical methods of analysis, 2d ed., (10) 305.
**Grim, R. E.,** modern concepts of clay materials, (3) 65; relation of composition to properties of clay, (3) 65.
**Grim, R. E., and Bradley, W. F.,** investigation of effect of heat on clay minerals, illite and montmorillonite, (3) 68, (9) 253.
**Grim, R. E., and Rowland, R. A.,** differential thermal analyses of clay minerals and other hydrous materials, (3) 68.
**Grunewald, M. E.,** systems CaO–$Fe_2O_3$ and MgO–$Fe_2O_3$, (7) 199.

**Haessler, C. G.,** solubility of ferric oxide in porcelain enamel ground coats, (2) 46.
**Hainbach, R.,** Pottery decorating, (2) 62.
**Hall, F. P.,** casting of clayware, a résumé, (4) 97; methods of measuring plasticity of clays, (4) 97.
**Hall, F. P., and Insley, Herbert,** compilations of phase-rule diagrams of interest to the ceramist and silicate technologist, (1) 24.
**Haller, Percy,** random notes on tank practice, (11) 350.
**Hansen, J. E.** (ed.), Manual of porcelain enameling, (4) 113; W. N. Noble cited on process control, (3) 90.
**Harkort, H.,** effect of particle size of quartz on properties of whiteware bodies, (1) 1; method of testing crazing of whiteware, (12) 359.
**Harman, C. G.,** clay technology as applied to enamels, (9) 255; laboratory experiments, 1939–1940, (4) 98; some characteristics of Ill. pottery clay, (4) 98.
**Harman, C. G., and Fraulini, F.,** properties of kaolinite as function of particle size, (9) 253.
**Harman, C. G., and Parmelee, C. W.,** testing and classification of ball clays, thermal history, (3) 72.
**Harrison, H. C.,** cyanite-clay refractories, I, (6) 181.
**Harrison, W. N.,** tentative tests for abrasion resistance, (12) 366.
**Hatch, T.,** determination of average particle size from screen analysis of nonuniform particulate substances, (1) 4.
**Hatch, T., and Choate, S. P.,** statistical description of size properties of nonuniform particulate substances, (1) 4.
**Hatschek, Emil,** general theory of viscosity of two-phase systems, (5) 151; Viskosität der Dispersoide, (5) 151.
**Hauser, E. A.,** Colloidal phenomena, (1) 6.
**Hauser, E. A., and LeBeau, D. S.,** studies in colloidal clays, II, (4) 116.
**Hauser, E. A., and Reed, C. E.,** studies in thixotropy, thixotropic behavior and structure of bentonite, II, (5) 151.
**Hecht, H.,** Textbook on ceramics, (2) 62.
**Hedvall, J. A.,** Reaktionsfähigkeit fester Stoffe, (7) 197, (10) 308.
**Hedvall, J. A., Alfredsson, S., Ohlsson, K., Runehagen, O., Waldenström, S., and Åkerström, A.,** reactivity and effects of lattice distortion in important glass-technical and ceramic systems, (10) 313.
**Hedvall, J. A., and Cohn, Gunther,** reactions in solid state, (10) 308.
**Hedvall, J. A., Garping, E., Lindekrantz, N., and Nelson, L.,** role of ions and crystallographic transformation in reactions in solid state, (10) 313.
**Hedvall, J. A., and Ny, V.,** double decomposition in solid state between synthetic or mineral ferrochromite and magnesium oxide or magnesium carbonate, (10) 313.
**Hedvall, J. A., and Sandberg, S. O.,** reactions in solid state of mixtures of active $Fe_2O_3$ and CaO, (10) 312.
**Henry, D. C.,** cataphoresis of suspended particles, equation of cataphoresis, I, (4) 117.
**Henry, E. C., and Taylor, N. W.,** acid and base binding capacities and viscosity relations in certain whiteware clays, (4) 98.
**Herman, A.,** factors influencing autoclave chemical durability tests of glass containers, (2) 53.
**Hermann, S.,** innovations in realm of silicon compounds, (10) 293.
**Heubach, A. R.,** light green chromium stains, (6) 169.
**Heusler, O.,** remarks on equilibrium during reduction of chromium sesquioxide and uranium dioxide by means of carbon and effect of nitrogen on uranium carbide, (10) 296.
**Hild, K.,** formation of spinel $Al_2ZnO_4$ by reaction in solid state, (10) 308.
**Hill, E. C.,** data on development of terra cotta glazes, (8) 234.
**Hillebrand, W. F., and Lundell, G. E. F.,** Applied inorganic analysis, (10) 205.
**Hillier, James,** electron microscope in determination of particle-size characteristics, (1) 2.
**Hodgman, C. D.,** Handbook of chemistry and physics, (1) 24.
**Hösgh, E. v., and Goerz, C. P.,** lens for optical purposes, (6) 193.
**Hoffman, M. K., and Barth, J. A.** (ed.), Dictionary of inorganic chemical reactions, (1) 24.
**Holland, A. J., and Preston, Eric,** microscopical examination and identification of crystalline products in commercial glasses, I–II, (11) 330.
**Holscher, H. H.,** effects of autoclave treatments on ceramic bodies and glazes, (12) 355; expansion of ceramic bodies caused by liquid and vapor penetration, (12) 355.
**Hopkinson, J.,** electrostatic capacity of glass, (2) 34.
**Hovestadt, H.,** Jena glass and its scientific and industrial applications, (2) 58, (4) 121, (11) 354.
**Howe, E. E., and Fellows, R. L.,** effect of manganese, nickel, and cobalt oxide on adherence and reboiling properties of ground-coat enamels, (11) 351.
**Hüttig, G. F., and Frantz, E.,** "memory" of solid materials, (7) 198.
**Hüttig, G. F., and Kölbl, F.,** active oxides, aluminum oxides and their addition compounds with water, LXVII, (7) 198.
**Hüttig, G. F., Tschakert, H. E., and Kittel, H.,** investigation on course of zinc ferrite formation from zinc oxide and iron oxide, (10) 314.
**Hüttig, G. F., Zinker, D., and Kittel, H.,** active stages encountered during transformation of mixture of metal oxides into chemical combination, (10) 314.
**Hugill, W.,** preparation and properties of some synthetic spinels, (7) 198.
**Hugill, W., and Green, A. T.,** some factors influencing durability of chrome-magnesite brick in basic open-hearth steelmaking furnaces, (7) 202.
**Huggins, M. L.,** density of silicate glasses as function of composition, (1) 11, (1) 13; dispersion of silicate glasses as function of composition, (1) 16; refractive index of silicate glasses as function of composition, (1) 15; silicate glasses, calculation of density, (9) 262.
**Huggins, M. L., and Sun, K.-H.,** calculation of density and optical constants of glass from composition in weight percentage, (1) 10, (1) 13, (9) 262; changes of properties of glass resulting from changes of composition, (1) 17.
**Huggins, M. L., Sun, K.-H., and Davis, D. O.,** dispersion of silicate glasses as function of composition, II, (1) 16.
**Hull, A. W., and Berger, E. E.,** glass-to-metal seals, (3) 88.
**Hunter, R. S.,** instructions for use of Hunter multipurpose reflectometer, (6) 165; multipurpose photoelectric reflectometer, (6) 166.
**Hurst, T. L., and Andrews, A. I.,** study of effect of soluble salts present in enamel mill liquors, (1) 25.
**Hutchison, Clark,** effect of various factors on abrasive resistance of porcelain enamels, (12) 366.

**Insley, Herbert,** determination of source and means of prevention of stones in glass, (11) 330; petrographic microscope as instrument for glass technologist, (11) 350; study of origin of stones in glass, (11) 330.
**International Critical Tables,** Vol. I, (11) 350; Vol. IV, (1) 24.
**Irwin, J. T., and Bevis, R. E.,** modification of the Bingham plastometer, (4) 98.

**Jander, W.,** reactions in solid state, course of reactions in solid state, II, (7) 198; reactions in solid state at high temperatures, tricalcium silicate, most important constituent of Portland cement clinker, XVIII, (10) 312.
**Jander, W., and Bunde, K.,** reactions in solid state at high temperatures, intermediate steps in formation of zinc aluminate from zinc oxide and aluminum oxide in solid state, XIV, (10) 315.
**Jander, W., and Hermann, H.,** reactions in solid state at high temperatures, theory of active form which appears at beginning of reaction in solid state, XXIII, (7) 198, (10) 314.
**Jander, W., and Hoffman, E.,** reactions in solid state at high temperatures, reaction between CaO and $SiO_2$, XI, (7) 198.
**Jebsen-Marwedel, H.,** chemistry of glass-furnace construction, slagging processes above metal line, I, (11) 350; influence of surface tension of slag glasses from refractory materials on their tendencies to form cords and streaks (5) 143; remarks on influence of surface tension in glassmelting processes, (5) 143.
**Jefferys, Harold,** draining of a vertical plate, (7) 203.
**Jeffrey, G. B.,** motion of ellipsoidal particles immersed in viscous fluid, (5) 152.
**Jenkins, F. A., and White, H. E.,** Fundamentals of physical optics (on Fresnal equations), (5) 145.
**Jenny, Hans, and Reitemeier, R. F.,** electrokinetic properties in relation to base-exchange phenomena, (4) 116.
**Jevons, W.,** band spectra of silicon oxide and chloride, and chlorides of carbon, boron, and aluminum, (10) 294; ultraviolet band-system of carbon monosulfide and its relation to those of carbon monoxide ("4th positive" bands) and silicon monoxide, (10) 294.
**Johannsen, Albert,** Manual of petrographic methods, (11) 330.
**Johnson, A. L., and Lawrence, W. G.,** fundamental study of clay, surface area and effect on exchange capacity of kaolinite, IV, (3) 77.
**Johnson, A. L., and Norton, F. H.,** fundamental study of clay: preparation of purified kaolinite suspension, I, (3) 77, (4) 117, (5) 158; mechanism of deflocculation in clay–water system, II, (4) 98.
**Johnson, P. D.,** suggestion for tetrahedral packing, (5) 153.
**Jost, Wilhelm,** Diffusion und chemische Reaktion in festen Stoffen, (7) 197, (10) 308.

**Joule, J. P.**, drift in ice-point readings, (2) 57.
**Judd, D. B., and Kelly, K. L.**, method of designating colors, (6) 165.
**Juran, J. M.**, A, B, C of quality control, (12) 377.

**Kautz, Karl**, discussion on Lord's paper, (2) 46.
**Keller, F.**, dielectric losses of glasses of different composition, (2) 33.
**Keller, H.**, cited on high-frequency materials, (6) 176; curves for variation of dissipation factor with frequency, (6) 177.
**Keller, W. D., P. C. E.** of coarse-grained refractories, (8) 241.
**Kelley, O. J., and Shaw, B. T.**, studies of clay particles with electron microscope, hydrodynamic considerations in relation to shape of particles, III, (3) 77.
**Kelley, W. P.**, modern clay researches in relation to agriculture, (4) 116.
**Kelley, W. P., and Jenny, H.**, relation of crystal structure to base exchange and its bearing on base exchange in soils, (1) 6.
**Keppeler, Gustav**, surface tension of glass, (6) 186.
**Kerr, P. F.**, decade of research on nature of clay, (9) 253.
**King, B. W., Jr.**, effect of particle size and index of refraction on reflectance, (9) 258.
**King, R. M.**, mechanics of enamel adherence: role of cobalt oxide in metal and oxide precipitation during ground-coat firing cycle and determination of temperature and time intervals of precipitation, XIV, (11) 350; influence of cobalt and nickel oxides on metal precipitation at ground coat-iron interface, XV, (11) 350.
**Klein, A.**, improved hydrometer for use in fineness determinations, (1) 2.
**Klemm, W., and Grimm, L.**, lower vanadium oxides, (10) 297.
**Klinefelter, T. A., and Meyer, W. W.**, properties of American kaolins and comparison with English china clays, (4) 97.
**Klinefelter, T. A., Meyer, W. W., and Vachuska, E. J.**, some properties of English china clay, (4) 97.
**Knapp, Oscar**, optical glass, (4) 121.
**Koch, E., and Wagner, Carl**, formation of $Ag_2HgI_4$ from AgI and $HgI_2$ by reaction in solid state, (10) 311.
**Koenig, C. J.**, low-temperature vitreous bodies, (12) 359; nepheline syenite in low-temperature vitreous ware, (12) 360.
**Koenig, C. J., and Watts, A. S.**, durability of tableware decorations, (2) 64.
**Koenig, J. H.**, particle-size distribution of ingredients versus body properties, (1) 2.
**Koenig, J. H., and Henderson, F. C.**, particle-size distribution of glazes, (1) 2.
**Körber, F., and Oelsen, W.**, reactions of chromium with acid slags, (10) 296.
**Kolthoff, I. M., and Carr, C. W.**, studies on aging and coprecipitation, distribution coefficient of arsenate between magnesium ammonium phosphate and solution, XXXVII, (10) 312.
**Kondo, S., and Suzuki, S.**, talc porcelain, (2) 39.
**Kondo, S., and Yamauchi, T.**, inversion of quartz to tridymite, IX–X, (10) 286.
**Konopicky, K., and Caesar, F.**, science of chrome ores, degree of oxidation of iron in chrome ores, III, (7) 202.
**Kozeny, Josef**, über Kapillare Leitung des Wassers im Boden (Aufstieg, Versickerung, und Anwendung auf die Bewässerung), (11) 319.
**Krause, Otto**, relationships between grain size of quartz and properties of hard porcelain, I–II, (1) 1.
**Kreidl, N. J.**, possibilities of strontia in ceramics, (8) 234.
**Kreimer, G. S.**, simultaneous reduction of oxides of iron, titanium, and columbium with aluminum, (10) 296.
**Kriegel, W. W.**, summary of occurrence, properties, and uses of vermiculite at Libby, Mont., (2) 50.
**Kubelka, P., and Munk, F.**, Beitrag zur Optik der Farbenstriche, (9) 258.

**Landolt, H., and Börnstein, R.**, Physikalisch-Chemische Tabellen, (1) 23.
**Lange, L. A.**, effect of sodium nitrite in cover enamels, (1) 25.
**Lange, N. A., and Forker, G. M.**, Handbook of chemistry and physics, (1) 24.
**Langmuir, I.**, dissociation of solids and kinetic interpretation of phase rule, (1) 4.
**Larsen, E. S.**, microscopic determination of nonopaque minerals, (11) 330.
**Latimer, W. M., and Hildebrand, J. H.**, Reference book of inorganic chemistry, (10) 294.
**Lea, F. M., and Desch, C. H.**, Chemistry of Portland cement and concrete, (7) 199.
**Lemaire, F. E.**, monoxide of silicon, (10) 293.
**Lester, W. R.**, influence of silica stones on strength of glass containers, (11) 350; use of polarizing microscope in factory control, (3) 85.
**Lewis, W. K., Squires, Lombard, and Broughton, Geoffrey**, Industrial chemistry of colloidal and amorphous materials, (5) 135.
**Lillie, H. R.**, expansion characteristics of Corning 96% silica glass No. 790, (10) 301; high-temperature viscosities of soda-silica glasses, (5) 129; Margule's method of measuring viscosities modified to give absolute values, (5) 129; measurement of absolute viscosity by use of concentric cylinders, (5) 129; viscosity of glass between strain point and melting temperature, (5) 129, (10) 301; viscosity measurements in glass, (5) 129.
**Lin, C.-Y.**, effect of various catalysts on conversion of quartz to cristobalite and tridymite at high temperatures, (10) 287.
**Lintz, E. H.**, use of talc and pyrophyllite in semivitreous dinnerware bodies, (12) 355.
**Littleton, J. T.**, method for measuring softening temperature of glasses, (5) 133.
**Littleton, J. T., and Morey, G. W.**, Electrical properties of glass, (2) 39.
**Löfquist, H.**, microscopic studies on slag inclusions in iron and steel, (10) 296.
**Loomis, G. A.**, grain size of whiteware clays as determined by Andreasen pipette, (1) 2, (9) 253; properties of clay casting slip, (4) 98.
**Lord, J. O.**, critical analysis of some statements and experiments on adherence of sheet-steel ground coats, (2) 46.

**Macey, H. H.**, clay-water relationships and internal mechanisms of drying, VI, (3) 79.
**Macey, H. H., and Clews, F. H.**, measurement of apparent porosity, (2) 35.
**Maclaurin, R. C.**, intensity of light reflected from transparent substances, (5) 145.
**MacMichael, R. F.**, testing clay, (4) 98.
**Maier, C. G.**, sponge chromium, (10) 296.
**Markert, F. S.**, function of manganese dioxide in ground coat, (11) 351.
**Marshall, C. E.**, Colloids in agriculture, (1) 24.
**Martin, Geoffrey, and Bowes, E. A.**, researches on theory of fine grinding, IX–X, (1) 4.
**Martin, S. W.**, determination of subsieve particle-size distributions by sedimentation methods, (1) 2.
**McAdams, W. H.**, Heat transmission, (2) 51.
**McCollom, P. C., and Danielson, R. R.**, effect of firing treatment on physical properties of dry-process enamels, (12) 366.
**McDowell, C. M., and Usher, F. L.**, viscosity and rigidity in suspensions of fine particles, aqueous suspensions, I, (5) 151; nonaqueous suspensions, II, (5) 151.
**McGee, E. N.**, silica cement, (8) 241.
**McIntyre, G. H., and Irwin, J. T.**, application of Gardner mobilometer to enamel-slip consistency studies and control, (4) 98.
**McMaster, Harold**, variations of refractive index of glass with time and temperature in annealing region, (9) 267.
**McVay, T. N., Galbreath, W. W., Jr., and Allen, Dan**, raw topaz as a high-temperature bond for domestic kyanite, (8) 241.
**McVay, T. N., and Wilson, Hewitt**, substitution of topaz, domestic kyanite, and synthetic mullite-corundum for India kyanite, I–III, (6) 185, (9) 275.
**Mellor, J. W.**, Comprehensive treatise on inorganic and theoretical chemistry, Part 2, carbon, silicates, Vol. VI, (10) 294; Vol. IX, (1) 24; crazing and peeling of glazes, (12) 359; discussion of C. E. Rameden, "notes on chromium glazes," (6) 174.
**Merwin, H. E.**, optical properties and theory of color of pigments and paints, (6) 168; system MgO–FeO–$Fe_2O_3$ in air at one atmosphere, (6) 173.
**Meyer, O.**, course of reactions between graphite and oxides, especially between heavy metallic carbides and oxide, (10) 296.
**Milbauer, J.**, action of oxygen on metallic oxides at high temperature and pressure, (6) 169.
**Minton, R. H.**, influence of some unusual zinc compounds in chrome green glaze, (6) 174.
**Mitchell, Lane, and Henry, E. C.**, nature of Ga. kaolins, I–II, (9) 253.
**Monack, A. J., and Beeton, E. E.**, analysis of strains and stress in glass, I–IV, (3) 87.
**Mooney, Melvin**, electrophoresis and the diffuse ionic layer, (4) 117.
**Morey, G. W.**, devitrification of soda-lime-silica glasses, (11) 344; effect of magnesia on devitrification of soda-lime-silica glass, (8) 222; Properties of glass, (4) 121, (9) 261, (11) 340.
**Morey, G. W., and Bowen, N. L.**, ternary system sodium metasilicate–calcium metasilicate–silica, (3) 83.
**Morgan, S. O.**, dielectric behavior of some ceramic materials, (2) 33.
**Mott, N. F., and Gurney, R. W.**, Electronic processes in ionic crystals, (10) 311.
**Moullin, E. B.**, molecular nature of a dielectric, (6) 175.
**Müller, H.**, theory of electrophoretic migration, (4) 116; zur allgemeinen Theorie der rachen Koagulation, (3) 77.
**Mulliken, R. S.**, isotope effect of band spectra, I, (10) 297; spectrum of boron monoxide, II, (10) 297.
**Murgatroyd, J. B.**, significance of surface marks on fractured glass, (3) 86.

**Nacken, R., and Grunewald, M. E.**, chemical combinations of lime and magnesia with ferric oxide, (7) 198.
**Nargund, K. S., and Watson, H. E.**, reactions of chromates at high temperatures, system lime–chromium oxide–oxygen, II, (6) 169.
**Noble, W. N.**, "process control" in Manual of porcelain enameling, (3) 90.
**Noda, Tokiti, and Hasegawa, Masotosi**, effect of addition of salt vapor on synthesis and crystal growth of spinel, (7) 198.
**Northrup, J. N., and Kunitz, M.**, improved type of electrocataphoresis cell, (4) 116.
**Norton, F. H.**, critical study of differential thermal method for identification of clay minerals, (3) 68; measuring viscosity of glass, (5) 129.
**Norton, F. H., and Speil, S.**, fractionation of clay into closely monodispersed systems, (3) 77, (4) 117; measurement of particle sizes in clay, (4) 117.
**Noyes, Bradford**, circuit for temperature controls, (2) 58.

**O'Meara, R. G., Norman, J. E., and Hammond, W. E.**, froth flotation and agglomerate tabling of feldspars, (1) 6.
**Orcel, Jean**, differential thermal analysis for determination of constituents of clays, laterites, and bauxites, (3) 68.
**Owens-Illinois Glass Co. General Research Laboratory**, effect of barium oxide and zinc oxide on properties of soda-dolomite lime-silica glass, (8) 221, (12) 370; effect of iron oxide on properties of soda-dolomite lime-silica glass, (8) 221.
**Owens, J. S., and Emanuel, E. C.**, effect of storage conditions on weathering of commercial glass containers, (5) 157.

**Palmer, W. E.**, influence of flint particle size on permanent moisture and thermal expansion in porous earthenware bodies, (12) 355.
**Pardee, J. T., Glass, J. J., and Stevens, R. E.**, massive low-fluorine topaz from Brewer Mine, S. C., (6) 181.
**Parmelee, C. W., and Ally, Abde**, some properties of chrome spinel, (7) 199.
**Parmelee, C. W., and Amberg, C. R.**, solubility of quartz and clay in feldspar, (2) 40.
**Parmelee, C. W., Badger, A. E., and Ballam, George**, study of group of typical spinels, (7) 198.
**Parmelee, C. W., and Harman, C. G.**, effect of alumina on surface tension of molten glass, (6) 186; surface tensions of molten glasses, surface tensions of soda-lime-silica melts, II, (6) 186, (7) 205.
**Parmelee, C. W., and Lyon, K. C.**, surface tensions of molten glasses, surface tension of molten glass, I, (6) 186.
**Parmelee, C. W., and Monack, A. J.**, devitrification of glass, relative stabilities of different glasses, time-temperature curves of

visible devitrification, and proposed classification of glasses, (11) 350.
**Parmelee, C. W., and Morgan, W. R.**, effect of fineness of flint on vitrification of a ceramic body, (1) 1.
**Parmelee, C. W., and Shaw, D. T.**, effect of additions of CaO and MgO on enamel glasses, (11) 354.
**Partridge, J. H.**, effect of gases on commercial glass containing lead oxide, (11) 350.
**Peck, A. B.**, new glass stone, $Na_2O \cdot 3CaO \cdot 6SiO_2$, (11) 330.
**Peddle, C. J.**, development of various types of glass, VI–IX, (11) 350; comparison of alkali-lime-silica and alkali-lead oxide-silica glasses, X, (11) 350.
**Percy, J.**, president's address, (10) 298.
**Peskin, W. L.**, effect of furnace atmosphere on colors derived from chromic oxide, (6) 165.
**Pfund, A. H.**, Brewsterian angle and refractive indices, (5) 147.
**Phelps, G. W.**, notes on casting, (4) 98.
**Piccolpasso, C.**, Potters art, (2) 62.
**Pierce, R. H. H., Jr., and Austin, J. B.**, comparison of thermal expansion of used silica brick from insulated and uninsulated open-hearth furnace roof, (10) 286.
**Pillai, D. S.**, effect of zinc oxide on coloring properties of chromium, cobalt, and iron oxides, (5) 174.
**Pole, G. R., and Taylor, N. W.**, kinetics of solid-phase reactions of certain carbonates with mullite, silica, and alumina, (7) 197.
**Porcelain Enamel Institute**, test for acid resistance of porcelain enamel, flatware, I, (12) 368; test for resistance of porcelain enamels to surface abrasion, (12) 367.
**Posnjak, E.**, crystal structures of magnesium, zinc, and cadmium ferrites, (6) 173.
**Potter, H. N.**, method of making silicon monoxide, (10) 293; Monox, (10) 293; process of producing silicon monoxide, (10) 293; production of "Monox," new electric furnace product, (10) 293; silicon monoxide, (10) 293.
**Potts, J. C.**, comparison of rates of melting glasses made from sodium carbonate, dolomite, limestone, and silica sand, (8) 225; melting rate of soda-lime-silica glasses as influenced by composition and effect of minor constituents, (8) 225.
**Preston, Eric**, crystallization relationship of soda-lime-magnesia-silica glass as used for drawn sheet and process of devitrification, (11) 350.
**Preston, Eric, and Turner, W. E. S.**, fundamental studies of glassmelting process, effect of particle size and temperature on rate of glass formation of pure soda-lime-silica glass, (11) 350.
**Preston, F. W.**, bottle breakage, causes and types of fractures, (3) 86; propagation of fissures in glass and other bodies with special reference to split-wave front, (3) 86; study of rupture of glass, (3) 86.
**Proctor, R. F., and Douglas, R. W.**, measurement of viscosity of glass at high temperatures by rotating cylinder viscometry, (5) 154.
**Purdy, R. C., and Brown, G. H.**, study of chromium tin pinks, (6) 174.

**Quincke, G.**, cited on electroosmotic flow of liquids in cell, (4) 116.

**Rait, J. R.**, viscosity of slags and glasses, I–II, (5) 130.
**Ramsden, C. E.**, study of chromium red glaze, (8) 234.
**Rankin, G. A., and Merwin, H. E.**, ternary system $MgO–Al_2O_3–SiO_2$, (7) 199, (11) 327.
**Rayleigh, Lord**, collected papers, (5) 145; polished surfaces of black glass, (5) 145.
**Rees, W. J.**, use of Indian sillimanite, (6) 181.
**Reimers, J. C.**, application of hydrometer method of fineness analysis to porcelain enamel slips, (1) 3.
**Richardson, E. G.**, some physical properties of clay, (5) 153.
**Riddle, F. H.**, andalusite as a refractory, (6) 181.
**Rigby, G. R., Dodd, A. E., White, R. P., and Green, A. T.**, investigation of permanent expansion of silica products containing cristobalite, (10) 287.
**Rigterink, M. D.**, improved ceramic dielectric materials, (2) 33, (10) 301.
**Rigterink, M. D., Grisdale, R. O., and Morgan, S. O.**, relation between chemical composition and dielectric properties of ceramic materials with low dielectric losses, (2) 39.
**Robinson, W. G.**, ceramic insulations for high-frequency work, (6) 176.
**Robinson, W. O.**, determination of organic matter in soils by hydrogen peroxide, (4) 101.
**Rodden, C. J.**, removal of static charges from glassware by ultraviolet light, (10) 303.
**Rogers, A. F., and Kerr, P. F.**, Thin section mineralogy, (1) 24, (3) 88; Optical mineralogy, 2d ed., (11) 350.
**Roller, P. S.**, classification of methods of mechanical analysis of particulate materials, (1) 2; law of size of distribution and statistical description of particulate materials, (1) 4.
**Roller, P. S., and Roundy, P. V., Jr.**, surface area of Portland cement, (1) 2.
**Rosenberg, J. E., and Langerman, A.**, method and apparatus for studying physical properties of vitreous enamels on steel, (11) 352; tearing of vitreous enamels, (1) 25.
**Russell, Ralston, Jr.**, development of superduty refractories from Ohio, Pa., and Ky. fire clays, (4) 112.

**Sands, J. W.**, Invar, Elinvar, and related iron-nickel alloys, (10) 304.
**Saper, P. G.**, rotational analysis of ultraviolet bands of silicon monoxide, (10) 294; ultraviolet bands of SiO, (10) 294.
**Satterly, John, and Givens, H.**, shape of liquid film draining on vertical, clean wetted glass plate and combined effects of gravity, viscosity, surface tension, and evaporation on same, III, (7) 203.
**Saunders, J. B.**, expansivity of a Vycor-brand glass, (10) 301.
**Sauveur, Albert**, Metallography and heat-treatment of iron and steel, (11) 336.
**Schade, W.**, photographic objective, (6) 194.
**Schober, Rolf, and Tilo, Erich**, chemical investigation of silicates, topax, $Al_2SiO_4$ (F, OH, O) and its synthesis and a new aluminum oxide containing fluorine, (6) 183.
**Scholes, S. R.**, rapid method for estimation of soda in glasses, (5) 148.
**Schott, E.**, high-frequency losses in glasses and other dielectrics, (2) 34.
**Schottky, W.**, mechanism of ionic motion in solid electrolytes, (10) 311.
**Schramm, Edward, and Hall, F. P.**, notes on casting slip, (4) 97.
**Schurecht, H. G.**, relation of salt to clay in purification of clays, (4) 98.
**Schurecht, H. G., and Pole, G. R.**, effect of water in expanding ceramic bodies of different compositions, (12) 355; methods for measuring strains between glazes and ceramic bodies, (8) 236, (12) 355; moisture expansion of glazes and other ceramic bodies, (12) 355.
**Schwarz, H.**, cited on effect of relative humidity on dissipation factor of insulating materials, (6) 177.
**Schweyer, H. E.**, particle-size studies, (1) 7.
**Schweyer, H. E., and Work, L. T.**, methods for determining particle-size distribution, (1) 2.
**Scott, W. W.**, Standard methods of chemical analysis, Vol. II, (10) 305.
**Sell, G. E.**, refractories from metallurgical viewpoint, (11) 327.
**Sell, G. E., and Staff**, orthosilicates of alkaline earths with special reference to their uses in the refractory field, (7) 198; bibliography, (7) 200.
**Shearer, W. L.**, control of plasticities of ceramic slips, (4) 98.
**Shewart, W. A.**, Economic control of quality of manufactured product, (12) 374.
**Silverman, W. B.**, effect of alumina on devitrification of soda-lime-silica glasses, (3) 81, (8) 221, (11) 340; effect of alumina on devitrification of sodium oxide-dolomite lime-silica glasses, (3) 83, (8) 221, (11) 340; effect of alumina on expansion and deformation temperatures of soda-lime-silica glasses, (8) 221; surface tension of glass and its effect on cords, (5) 143, (6) 186.
**Simon, L. E.**, Engineer's manual of statistical methods, (12) 374.
**Small, G. G., Brooksbank, R. J., and Thornton, W. M.**, electrical resistance of moisture films on glazed surfaces, (6) 180.
**Smoluchowski, M.**, Handbuch der Electricität und des Magnetisimus, (4) 116.
**Smyth, C. P.**, Dielectric constant and molecular structure, (6) 175.
**Sosman, R. B.**, properties of silica, (10) 286, (10) 298, (10) 302.
**Soyck, Wener**, Eigentümlichkeiten des dielektrischen Verhaltens rutilhaltiger Kondensator Baustoffe, (6) 179.
**Speil, S.**, effect of adsorbed electrolytes on properties of monodisperse clay–water systems, (4) 98.
**Spencer, S. D.**, do glass rods and tubes bend under their own weight, (11) 352.
**Sproat, I. E., Treischel, C. C., and Emrich, E. W.**, all-American sanitary-ware body, (4) 98; use of organic dispersing agents in casting slips, (4) 98.
**Starke, K.**, surface of catalytic mixtures and surface changes caused by reaction in solid state, (10) 315.
**Stott, V. H., Turner, D., and Sloman, H. A.**, effect of thermal treatment of glass by precise viscometry, (7) 203.
**Stuckert, L.**, stannic oxide as opacifier in wet enamels, (4) 113.
**Stuckey, J. L., and Amero, J. J.**, physical properties of massive topaz, (6) 181.
**Swicker, V. C.**, ring-section examination of glass containers, (3) 85.

**Tammann, G.**, transformations in crystals with lattice defects, (7) 198.
**Taylor, N. W.**, elastic aftereffects and dielectric absorption in glass, (2) 60; law of annealing of glass, quantitative treatment and theoretical interpretation, (2) 58; reactions in solids in absence of liquid phase, (7) 197.
**Taylor, N. W., and Dear, P. S.**, elastic and viscous properties of several soda-silica glasses in annealing range of temperature, (5) 129.
**Taylor, N. W., and Lin, C.-Y.**, effect of various catalysts on conversion of quartz to cristobalite and tridymite at high temperatures, (10) 286, (11) 350.
**Taylor, N. W., McNamara, E. P., and Sherman, J.**, elastico-viscous properties of soda-lime-silica glass at temperatures near "transformation point," (5) 129.
**Taylor, N. W., and Noyes, Bradford, Jr.**, aging of thermometers, (9) 267.
**Thiemecke, H.**, thermal and moisture expansion of ball clays and bodies at different temperatures, (12) 355; thermal and moisture expansion of kaolins and bodies at different temperatures, (12) 355.
**Thompson, C. L.**, identification of stones in glass, (11) 330.
**Thoreen, R. C.**, comments on hydrometer method of mechanical analysis, (1) 6.
**Thurnauer, Hans**, review of ceramic materials for high-frequency insulation, (2) 33.
**Thurnauer, Hans, and Badger, A. E.**, dielectric loss of glass at high frequencies, (2) 36.
**Thurnauer, Hans, and Rodriguez, A. R.**, notes on constitution of steatite, (2) 35.
**Tiede, R. L., and Tooley, F. V.**, effect of temperature on homogenizing rate of soda-lime-silica glass, (9) 264.
**Tool, A. Q., and Insley, Herbert**, crystalline silica in certain devitrified glasses, (10) 286.
**Tooley, F. V., and Greene, J. F.**, personal communication, (10) 305.
**Tooley, F. V., and Tiede, R. L.**, factors affecting degree of homogeneity of glass, (9) 264.
**Tone, F. J.**, production of silicon in electric furnace, (10) 293.
**Trostel, L. J.**, absorption of magnesia by chromite spinel, (7) 202.
**Turnbull, J. C.**, separation of inhomogeneities in bottle glass by density differences, (2) 42, (9) 263.
**Turnbull, J. C., and Ghering, L. G.**, cord analysis, (2) 42, (9) 263.

**U. S. Ordnance Dept.**, Document No. 2037, manufacture of optical glass and optical systems, (11) 352.

**Van Nieuwenburg, C. J., and Zijlstra, H. J.**, dilatometric measurements on tridymite and cristobalite, (10) 286.
**Vasenin, I. F.**, reciprocal action of CaO and $Cr_2O_3$ on heating, (6) 169.
**Vaughan, T. C.**, determination of density by flotation method, (9) 263.

**Wagner, Carl,** mechanism of formation of ionic compounds of high order (double salts, spinels, silicates), (10) 310; theory of arranged mixed phases, disarranged phenomena in polar compounds as basis for ionic and electronic conduction, III, (10) 311; theory of tarnishing process, (7) 198; theory of tarnishing process, II, (10) 311.
**Wagner, Carl, and Schottky, W.,** theory of arranged mixed phases, I–II, (10) 311.
**Washburn, E. W., and Shelton, G. R.,** viscosities and surface tensions of soda-lime-silica glasses at high temperatures, viscosities of glasses at high temperatures, I, (5) 134.
**Waterton, S. C.,** viscous-temperature relation and some inferences on nature of molten and plastic glass, (5) 138.
**Watkins, R. T.,** degree to which different glaze compositions take vapor lusters, (2) 64.
**Watts, A. P.,** effect of furnace atmospheres on quality of certain types of glazes, (6) 165.
**Watts, A. S.,** citation of unpublished work on $SrO$–$Al_2O_3$–$SiO_2$ system, (8) 234.
**Weber, R.,** über den Einfluss der Zusammensetzung des Glases auf die Veranderungen des Nullpunkts, (2) 58.
**Weil, R.,** microscopic study of alpha-beta transformation of natural cristobalite, (10) 286.
**Weiser, H. B.,** Colloid chemistry, (1) 24.
**Weltmann, R. N., and Green, H.,** rheological properties of colloidal solutions, pigment suspensions, and oil mixtures, (5) 151.
**Westman, A. E. R.,** effect of mechanical pressure on imbibitional and drying properties of some ceramic clays, I, (3) 79.
**Whitesell, R. J.,** effect of soluble salts on properties of enamels, (1) 25.
**Whitney, W. R.,** General Electric Co., personal communication, (10) 294.
**Whittaker, H.,** effect of particle size on plasticity of kaolinite, (3) 77.
**Wiberg, E.,** "borohydrates" and boron suboxides of Travers, Ray, and Gupta, (10) 297.
**Wichers, Edward, Finn, A. N., and Clabaugh, W. S.,** comparative tests on chemical glassware, (10) 303.
**Wiebe, H. F.,** über die Herstellung von Normalglas für Thermometer, (2) 58.
**Wilde, W. T., and Rees, W. J.,** ternary system $MgO$–$Al_2O_3$–$SiO_2$, (11) 327.
**Williamson, E. D., and Adams, L. H.,** temperature distribution in solids during heating or cooling, (9) 272.
**Wills, G. A.,** notes on casting slip in production, (4) 97.
**Wilson, G. V.,** notes on formation of certain rockforming minerals in and about glass furnaces, (11) 330.
**Wilson, Hewitt,** Ceramics-clay technology, (1) 24.
**Wilson, Hewitt, and Page, G. A.,** simple dispersion test for clays, (4) 98.
**Winchell, A. N.,** Elements of optical mineralogy, Parts I and II, (11) 330; Microscopic characters of artificial inorganic solid substances or artificial minerals, (7) 199; Optic and microscopic characters of artificial minerals, (1) 24.
**Winkler, C.,** reduction of oxides by magnesium, (10) 293.
**Winter, Aniuta,** structural homogeneity of glass, (9) 266; transformation region of glass, (9) 266.
**Winterkorn, H. F.,** condition of water in porous systems, (3) 80.
**Woods, L. H.,** silicon oxide bands, (10) 294.
**Wretblad, P. E.,** X-ray studies on systems $Fe_2O_3$–$Cr_2O_3$ and $Fe_2O_3$–$Mn_2O_3$, (6) 173.
**Wright, E. P.,** study of absorption of two earthenware bodies and their resistance to crazing in steam test, (12) 355.

**Young, J. C.,** density data, (1) 13.
**Young, J. C., and Finn, A. N.,** comparison of two recent studies on refraction and dispersion of glasses, (1) 15; effect of composition and other factors on specific refraction and dispersion of glasses, (1) 15.

**Zapffe, C. A., and Sims, C. E.,** silicon monoxide, (10) 297; silicon-oxygen equilibria in liquid iron, (10) 297.
**Zarbo, G. J., Hunt, H. J., and Smith, D. C.,** analysis of cords and stones, (9) 264.
**Zintl, W., Bräuning, W., Grube, H. L., Krings, W., and Morawietz, W.,** silicon monoxide, (10) 295.
**Zintl, E., and Grube, H.,** reduction of inorganic oxidic compounds by silicon monoxide, (10) 295.
**Zintl, W., Krings, W., and Bräuning, W.,** reduction of inorganic compounds by silicon monoxide, (10) 295.
**Zintl, E., Morawietz, W., and Gantinger, E.,** boron monoxide, (10) 297.
**Zschacke, F. H.,** deposits in heat regenerators and waste-gas channels, (10) 297; volatilization of components of glass during melting and fining, (10) 297.

# CORRECTIONS

## EFFECT OF GLASS ON ALCOHOLIC PRODUCTS STORED IN BOTTLES AT ROOM TEMPERATURE FOR THREE YEARS

By A. Herman and H. L. Shay

Published in *Jour. Amer. Ceram. Soc.*, **27** [2] 53–57 (1944).

The words "decrease" or "decreased" should be substituted for "increase" or "increased" in the following lines:

(1) p. 53 (Abstract), lines 5–6; line 7.
(2) p. 54, Figs. 1 and 2; left column, line 4.
(3) p. 55, Figs. 3 and 4.
(4) p. 56, left column, last paragraph, line 1 and lines 5 and 6.

## PARTICLE-SIZE DISTRIBUTION OF STEATITE TALC

By J. S. White

Published *Jour. Amer. Ceram. Soc.*, **27** [11] 320–23 (1944).

Page 323, Fig. 6: The designations "% St'd" and "% Fine" on the scales at the bottom of the graph should be transposed so that the upper scale is designated "% Fine" and the lower "% St'd." The curves will then indicate that the shrinkage is higher when the ultra-fine talc is used, which is actually the case.

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of

# The American Ceramic Society

A Monthly Journal Devoted to the
Arts and Sciences Related to
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Volume 27, 1944

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# TABLE OF CONTENTS

for

Volume 27, 1944


**January [No. 1], pp. 1–31**

1. R. Russell, Jr., and R. S. Weisz: Some Particulate Characteristics of Commercial Flint and Feldspar ..... 1–7
2. M. F. Goudge: Brucite Magnesia ..... 8–10
3. M. L. Huggins and K.-H. Sun: Changes of Properties of Glass Resulting from Changes of Composition ..... 10–12
4. M. L. Huggins and K.-H. Sun: Glass Properties and the Periodic Table ..... 13–17
5. M. K. Blanchard and A. I. Andrews: Fundamental Properties of Soluble Salts in Enamel Mill Liquors ..... 17–24
6. M. K. Blanchard and A. I. Andrews: Enamel Film Strength as Affected by Properties of Enamel Before, During, and After Drying ..... 25–31

**February [No. 2], pp. 33–64**

7. H. F. G. Ueltz: Crystalline and Glassy Phases of Steatite Dielectrics ..... 33–39
8. A. G. Johnson and D. B. Underhill: Quantitative Determination of Glassy Phase in a Ceramic Body ..... 40–41
9. F. V. Tooley and R. L. Tiede: Factors Affecting Degree of Homogeneity of Glass ..... 42–45
10. A. I. Andrews and H. R. Swift: Effect of Composition and Temperature on Solubility of Iron Oxide in Ground-Coat Enamel Glass ..... 46–50
11. F. K. Pence and E. B. Blount: Vermiculite as Raw Material in Ceramic Manufacture ..... 50–52
12. A. Herman and H. L. Shay: Effect of Glass on Alcoholic Products Stored in Bottles at Room Temperature for Three Years ..... 53–57
13. N. W. Taylor and Bradford Noyes, Jr.: Aging Thermometers ..... 57–62
14. Forrest Burnham and C. M. Harder: Practical Production of One-Fire Luster Glazed Pottery ..... 62–64

**March [No. 3], pp. 65–95**

15. R. E. Grim and R. A. Rowland: Differential Thermal Analysis of Clays and Shales, Control and Prospecting Method ..... 65–76
16. F. H. Norton and A. L. Johnson: Fundamental Study of Clay: V, Nature of Water Film in Plastic Clay ..... 77–80
17. J. F. White and W. B. Silverman: Devitrification Characteristics of Alkali-Lead Oxide-Dolomite Lime-Silica Glasses ..... 81–85
18. Glass Container Association of America: Polariscopic Examination of Glass Container Sections ..... 85–89
19. E. C. Seabright: Methods for Testing Cleaner Solutions ..... 90–95

**April [No. 4], pp. 97–128**

20. Ralston Russell, Jr., and W. C. Mohr: Casting Characteristics of Clays: I, Improved Methods for Determination ..... 97–113
21. F. A. Petersen and A. I. Andrews: Enameled Utensil Manufacturer's Council Tentative Standard Solubility Tests for Porcelain Enameled Cooking Utensils ..... 113–15
22. W. L. Coughanour and J. L. Utter: Cataphoresis of Purified, Fractionated Kaolinite Particles ..... 116–20
23. W. A. Fraser and L. O. Upton: Optical Fluor-Crown Glasses ..... 121–28

**May [No. 5], pp. 129–64**

24. H. A. Robinson and C. A. Peterson: Viscosity of Recent Container Glass ..... 129–38
25. G. J. Zarbo, H. J. Hunt, and D. C. Smith: Analysis of Cords and Stones ..... 138–42
26. M. A. Knight: Surface Tension and Persistence of Cords in Glass ..... 143–45
27. F. L. Bishop, Jr.: Surface Layer of Sheet Glass ..... 145–48
28. S. R. Scholes and E. deR. Falcão: Rapid Method for Estimating Soda in Glasses, II ..... 148
29. F. H. Norton, A. L. Johnson, and W. G. Lawrence: Fundamental Study of Clay: VI, Flow Properties of Kaolinite-Water Suspensions ..... 149–64

**June [No. 6], pp. 165–95**

30. W. F. McDevit: Effect of Firing Atmosphere on Development of Colors in Ceramic Glazes ..... 165–75
31. H. H. Hausner: Influence of Humidity on Dielectric Properties of High-Frequency Ceramics ..... 175–81
32. G. R. Pole: Calcination of Topaz Ore in Rotary Kiln ..... 181–85
33. K. C. Lyon: Calculation of Surface Tensions of Glasses ..... 186–89
34. R. Kingslake: Optical Glass from Viewpoint of Lens Designer ..... 189–95

**July [No. 7], pp. 197–220**

35. V. D. Fréchette and A. I. Andrews: Investigation of Reactions of Simple Magnesia Spinels with Alkaline Earth Orthosilicates in Solid State ..... 197–202
36. C. G. Harman and C. F. Schaefer: Study of Factors Involved in Glaze-Slip Control: I, Glaze-Slip Specifications ..... 202–207
37. C. G. Harman and M. K. Blanchard: II, Correlation of Glaze Thickness with Air Permeability ..... 207–209
38. C. G. Harman and H. C. Johnson: III, Additional Glazes and Improved Technique and Apparatus ..... 209–14
39. C. G. Harman and H. C. Johnson: IV, Practical Uses for Coherence Value and Receptivity Measurements ..... 214–20

**August [No. 8], pp. 221–49**

40. Owens-Illinois Glass Company, General Research Laboratory: Effect of Substituting MgO for CaO on Properties of Typical Soda-Lime Glasses ..... 221–25
41. J. C. Potts, George Brookover, and O. G. Burch: Melting Rate of Soda-Lime Glasses as Influenced by Grain Sizes of Raw Materials and Additions of Cullet ..... 225–31
42. C. G. Harman: Suggestions for Solution of Difficult Glaze-Fit Problems ..... 231–33
43. E. S. McCutchen: Strontia and Its Properties in Glazes ..... 233–38
44. R. E. Birch and M. P. Hall: Some Factors Affecting P. C. E. of Silica Fire Clay ..... 239–41
45. W. W. Galbreath, Jr., T. N. McVay, and Dan Allen: Lightweight Superduty Kyanite-Topaz Refractory ..... 241–46
46. J. I. Tracey, Jr.: High-Alumina Clays of Pulaski and Saline Counties, Arkansas ..... 246–49

**September [No. 9], pp. 253–84**

47. B. W. King, Jr., H. D. Carter, and H. C. Draker: Properties of Enamel Slips: I, General Properties of Clays and Enamel Slips ..... 253–60
48. L. G. Ghering and M. A. Knight: Properties and Diagnosis of Cords in Pressed and Blown Glassware ..... 260–66
49. Aniuta Winter: Time Problem in Annealing ..... 266–74
50. T. N. McVay, W. W. Galbreath, Jr., and Dan Allen: Substitution of Topaz, Domestic Kyanite, and Synthetic Mullite-Corundum for India Kyanite: IV, Raw Topaz as High-Temperature Bond for Domestic Kyanite ..... 275–82
51. Edward Schramm: Glost Kiln Refractories ..... 282–84

**October [No. 10], pp. 285–316**

52. M. D. Beals and Samuel Zerfoss: Volume Change Attending Low-to-High Inversion of Cristobalite ..... 285–92
53. C. A. Zapffe: Conversion of Certain Refractory Oxides to Suboxide Form at High Temperatures ..... 293–98
54. M. E. Nordberg: Properties of Some Vycor-Brand Glasses ..... 299–305
55. H. J. Hunt: Analysis of Batch for Segregation ..... 305–308
56. Gunther Cohn: Significance of Topochemical Factors on Rate of Reactions Between Solid Salts ..... 308–16

**November [No. 11], pp. 317–54**

57. R. S. Lamar: Particle Shape and Differential Shrinkage of Steatite-Talc Bodies ..... 317–20
58. J. S. White: Particle-Size Distribution of Steatite Talc ..... 320–23
59. C. R. Amberg: Effect of Additions of Diopside on Properties of Wall-Tile Body ..... 324–26
60. H. N. Baumann, Jr.: Fusion Experiments in System $MgO$–$FeO$–$Cr_2O_3$–$Al_2O_3$ ..... 327–29
61. W. L. Fabianic: Glasshouse Stones ..... 330–50
62. R. M. King: Mechanics of Enamel Adherence: XVI, Influence of Manganese Dioxide on Metal Precipitation at Ground Coat-Iron Interface ..... 350–51
63. W. G. Martin and F. W. Lauck: Observations Indicating Absence of Plastic Flow in Glass Coating on Steel ..... 352–54

**December [No. 12], pp. 355–87**

64. H. Thiemecke: Notes on Moisture Expansion ..... 355–58
65. John Marquis: Cone 01 Glazes for Low-Temperature Vitreous Ware ..... 358–66
66. Clark Hutchison: Abrasion Resistance of Porcelain Enamels ..... 366–69
67. Owens-Illinois Glass Company, General Research Laboratory: Effect of Fluorine and Phosphorus Pentoxide on Properties of Soda-Dolomite Lime-Silica Glass ..... 369–72
68. L. G. Ghering: Refined Method of Control of Cordiness and Workability of Glass During Production, Statistical Control Charts Applied to Daily Measurements of Density ..... 373–87

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